USEPA CONTRACT LABORATORY PROGRAM

STATEMENT OF WORK

FOR

INORGANIC ANALYSIS

Multi-Media, Multi-Concentration

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STATEMENT OF WORK

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EXHIBIT A SUMMARY OF REQUIREMENTS

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Exhibit A - Summary of Requirements

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1.0 PURPOSE

The purpose of the multi-media, multi-concentration inorganic analytical service is to provide analytical data for use by the U.S. Environmental Protection Agency (USEPA) in support of the investigation and clean-up activities under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA) and the Superfund Amendments and Reauthorization Act of 1986 (SARA). Other USEPA Program Offices that have similar analytical data needs also use this service.

2.0 DESCRIPTION OF SERVICE

The inorganic analytical service provides a contractual framework for laboratories. This framework applies USEPA Contract Laboratory Program (CLP) analytical methods for the isolation, detection, and quantitative measurement of 23 metals (including mercury) and cyanide in water/aqueous and/or soil/sediment samples. The analytical service contract provides specific contractual requirements by which USEPA will evaluate the data.

3.0 DATA USES

This analytical service contract provides data which USEPA uses for a variety of purposes, such as: determining the nature and extent of contamination at a hazardous waste site, assessing priorities for response based on risks to human health and the environment, determining appropriate cleanup actions, and determining when remedial actions are complete. The data may be used in all stages in the investigation of hazardous waste sites, including: site inspections, Hazard Ranking System (HRS) scoring, remedial investigation/feasibility studies, remedial design, treatability studies, and removal actions.

The data may also be used in litigation against Potentially Responsible Parties in the enforcement of Superfund legislation. As a result, the Contractor must be aware of the importance of maintaining the integrity of the data generated under this contract, since it is used to make major decisions regarding public health and environmental welfare. The Contractor may be required to appear and testify to the accuracy and/or validity of the data generated.

4.0 SUMMARY OF REQUIREMENTS

4.1 Introduction to the Inorganic Statement of Work

The Statement of Work (SOW) is comprised of eight exhibits and two appendices. Exhibit A provides an overview of the SOW and its general requirements. Exhibit B contains a description of the reporting and deliverables requirements, in addition to the data reporting forms and instructions. Exhibit C specifies the Inorganic Target Analyte List (TAL) for this SOW with the Contract Required Quantitation Limits (CRQLs) for the sample matrices. Exhibit D details the required analytical procedures to be used with this SOW and resulting contracts. Exhibit E provides descriptions of required Quality Assurance/Quality Control (QA/QC), Standard Operating Procedures (SOPs), QA/QC performance, and the reporting of data. Exhibit F contains chain-ofcustody and sample documentation requirements. To ensure proper understanding of the terms utilized in this SOW, a glossary can be found in Exhibit G. When a term is used in the text without explanation, the glossary meaning shall be applicable. Specifications for reporting data in computer-readable format appear in Exhibit H. Appendix A provides examples of the data format requirements specified in Exhibit H. Appendix B contains a description of the requirements for performing

Exhibit A -- Section 4
Summary of Requirements (Con't)

modified analyses, as well as the analytical procedure for Graphite Furnace Atomic Absorption (GFAA).

4.2 Overview of Major Task Areas

For each sample, the Contractor shall perform the tasks described in each section. Specific requirements for each task are detailed in the exhibits referenced in the following sections.

- 4.2.1 Task I: Sample Receiving, Storage, and Disposal
- 4.2.1.1 Chain-of-Custody

The Contractor shall receive and maintain samples under proper chain-of-custody. All associated document control and inventory procedures shall be developed and followed. Documentation described herein shall be required to show that all procedures are strictly followed. This documentation shall be reported as the Complete Sample Delivery Group (SDG) File (CSF) (see Exhibit B). The Contractor shall establish and use appropriate procedures to safeguard confidential information received from USEPA.

4.2.1.2 Sample Scheduling/Shipments

Sample shipments to the Contractor's facility will be scheduled and coordinated by the Contract Laboratory Program (CLP) Sample Management Office (SMO). USEPA may request analyses that include all or a subset of the Inorganic Target Analytes listed in Exhibit C. The Contractor shall communicate with SMO personnel by telephone as necessary throughout the process of sample scheduling, shipment, analysis, and data reporting, to ensure that samples are properly processed.

- 4.2.1.2.1 Samples will be shipped routinely to the Contractor through an overnight delivery service. However, as necessary, the Contractor shall be responsible for any handling or processing of the receipt of sample shipments. This includes the pick-up of samples at the nearest servicing airport, bus station, or other carrier within the Contractor's geographical area. The Contractor shall be available to receive and process sample shipments at any time the delivery service is operating, including Saturdays, to ensure that short sample analysis time requirements can be met.
- 4.2.1.2.2 If there are problems with the samples (e.g., mixed media, containers broken or leaking) or sample documentation and paperwork (e.g., Traffic Reports/Chain of Custody Records not with shipment, sample and Traffic Report/Chain of Custody Record do not correspond), the Contractor shall immediately contact SMO for resolution. The Contractor shall immediately notify SMO and the USEPA Regional CLP Project Officer (CLP PO) regarding any problems and laboratory conditions that affect the timeliness of analyses and data reporting. In particular, the Contractor shall immediately notify SMO personnel and the USEPA Regional CLP PO in advance regarding sample data that will be delivered late and shall specify the estimated delivery date.
- 4.2.1.2.3 To monitor the temperature of the sample shipping cooler more effectively, each USEPA Regional Office may include a sample shipping cooler temperature blank with each cooler shipped. The temperature blank will be clearly labeled: USEPA COOLER

TEMPERATURE INDICATOR. The Contractor shall record the presence or absence of the cooler temperature indicator bottle on Form DC-1, Item 8 - Cooler Temperature Indicator Bottle (see Exhibit B).

- 4.2.1.2.3.1 When the USEPA Regional Office supplies a cooler temperature indicator bottle in the sample shipping cooler, the Contractor shall use the USEPA supplied cooler temperature indicator bottle to determine the cooler temperature. The temperature of the cooler shall be measured at the time of sample receipt by the Contractor.
- 4.2.1.2.3.2 The temperature of the sample shipping cooler shall be measured and recorded immediately upon opening the cooler.
- 4.2.1.2.3.3 To determine the temperature of the cooler: the Contractor shall locate the cooler temperature indicator bottle in the sample shipping cooler, remove the cap, and insert a calibrated thermometer into the cooler temperature indicator bottle. Prior to recording the temperature, the Contractor shall allow a minimum of 3 minutes, but not greater than 5 minutes, for the thermometer to equilibrate with the liquid in the bottle. At a minimum, the calibrated thermometer $(\pm 1^{\circ}C)$ shall have a measurable range of 0-50°C. Other devices which can measure temperature may be used if they can be calibrated to $\pm 1\,^{\circ}\text{C}$ and have a range of 0-50 $^{\circ}\text{C}$. If a temperature indicator bottle is not present in the cooler, an alternative means of determining cooler temperature shall be used. Under no circumstances shall a thermometer or any other device be inserted into a sample bottle for the purpose of determining cooler temperature. The Contractor shall contact SMO and inform them that a temperature indicator bottle was not present in the cooler. The Contractor shall document the alternative technique used to determine cooler temperature in the SDG Narrative.
- 4.2.1.2.3.4 If the temperature of the sample shipping cooler's temperature indicator exceeds 10°C, the Contractor shall contact SMO and inform them of the temperature deviation. SMO will contact the Region from which the samples were shipped for instruction on how to proceed. The Region will either require that no sample analysis(es) be performed or that the Contractor proceed with the analysis(es). SMO will in turn notify the Contractor of the Region's decision. The Contractor shall document the Region's decision and the EPA sample numbers of all samples for which temperatures exceeded 10°C in the SDG Narrative.
- 4.2.1.2.3.5 The Contractor shall record the temperature of the cooler on Form DC-1, under Item 9 Cooler Temperature, and in the SDG Narrative (see Exhibit B).
- 4.2.1.2.4 The Contractor is required to retain unused sample volume, used sample containers, and empty sample bottle containers for a period of 60 days after data submission. From time of receipt until analysis, the Contractor shall maintain <u>all</u> water/aqueous (preserved and unpreserved) and/or soil/sediment samples at 4°C (±2°C) (see Exhibit B).
- 4.2.1.2.5 The Contractor shall be required to routinely return sample shipping containers (e.g., coolers) to the appropriate sampling

office within 14 calendar days following shipment receipt (see contract, Section G titled, "Government Furnished Samples").

- 4.2.1.2.6 Sample analyses will be scheduled by groups of samples, each defined as a Case and identified by a unique EPA Case number assigned by SMO. A Case signifies a group of samples collected at one site or geographical area over a finite time period, and will include one or more field samples with associated blanks. Samples may be shipped to the Contractor in a single shipment or multiple shipments over a period of time, depending on the size of the Case.
- 4.2.1.2.6.1 A Case consists of one or more SDGs. An SDG is defined by the following, whichever is most frequent:
 - Each Case of field samples received, or
 - Each 20 field samples [excluding Performance Evaluation (PE) samples] within a Case, or
 - Each 7 calendar day period (3 calendar day period for 7 day turnaround) during which field samples in a Case are received (said period beginning with the receipt of the first sample in the SDG).
 - In addition, all samples and/or sample fractions assigned to an SDG must have been scheduled under the same contractual turnaround time. Preliminary Results have no impact on defining the SDG.
- 4.2.1.2.6.2 Samples may be assigned to SDGs by matrix (i.e., all soils in one SDG, all waters in another), at the discretion of the laboratory. However, PE samples received within a Case shall be assigned to an SDG containing field samples for that Case. Such assignment shall be made at the time the samples are received, and shall not be made retroactively.
- 4.2.1.2.6.3 Each sample received by the Contractor will be labeled with an EPA sample number, and accompanied by a Traffic Report/Chain of Custody Record bearing the sample number and descriptive information regarding the sample. EPA sample numbers are six digits in length. If the Contractor receives a sample number of any other length, the Contractor shall contact SMO immediately. The Contractor shall complete and sign the Traffic Report/Chain of Custody Record, recording the date of sample receipt and sample condition on receipt for each sample container. $\underline{\mbox{The}}$ Contractor shall also follow the instructions given on the Traffic Report/Chain of Custody Record in choosing the Quality Control (QC) samples when such information is provided. If no QC sample is designated on the Traffic Report/Chain of Custody Record, the Contractor shall select a sample and notify SMO for Regional acceptance. SMO shall contact the Region for confirmation immediately after notification.
- 4.2.1.2.6.4 The Contractor shall submit signed copies of Traffic Reports/Chain of Custody Records for all samples in a SDG to SMO within **three working days** following receipt of the last sample in the SDG. Faxed copies of Traffic Reports/Chain of Custody Records do not meet this requirement. Traffic Reports/Chain of Custody Records shall be submitted in SDG

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sets (i.e., all Traffic Reports/Chain of Custody Records for a SDG shall be clipped together) with an SDG Cover Sheet containing information regarding the SDG, as specified in Exhibit B.

- 4.2.1.2.6.5 EPA Case numbers, SDG numbers, and EPA sample numbers shall be used by the Contractor in identifying samples received under this contract both verbally and in reports/correspondence.
- 4.2.1.3 Modified Analysis

The Contractor may be requested by USEPA to perform modified analyses. These modifications will be within the scope of this SOW and may include, but are not limited to, analysis of additional analytes and/or lower quantitation limits. These requests will be made by the USEPA Regional CLP PO, USEPA Office of Superfund Remediation and Technology Innovation (OSRTI) Analytical Services Branch Inorganic Program Manager (ASB PM), and Contracting Officer (CO) in writing, prior to sample scheduling. If the Contractor voluntarily elects to perform these modified analyses, these analyses will be performed with no increase in per sample price. All contract requirements specified in the SOW/Specifications will remain in effect unless the USEPA CO provides written approval for the modification(s) and a waiver for associated defects. The USEPA CO approval must be obtained prior to sample scheduling.

- 4.2.2 Task II: Sample Preparation and Analysis
- 4.2.2.1 Overview

The Contractor is advised that the samples received under this contract are usually from known or suspected hazardous waste sites and may contain high (greater than 15%) levels of organic and inorganic materials of a potentially hazardous nature and of unknown structure and concentration, and should be handled throughout the analysis with appropriate caution. It is the Contractor's responsibility to take all necessary measures to ensure laboratory safety.

4.2.2.2 The Contractor shall prepare and analyze samples as described in Exhibit D. Sample preparation methods shall remain consistent for all samples analyzed within a Case. Prior to sample analysis, the Contractor shall review the Traffic Report/Chain of Custody Record for any special sample analysis instructions. Anomalies that occur during sample analysis shall be reported to SMO and the USEPA Regional CLP PO immediately.

The Contractor shall collectively review all analytical results associated with a sample. This includes undiluted, diluted, serial dilution, and interference results. The Contractor shall report any significant anomalies between these results in the SDG Narrative indicating possible matrix interferences.

- 4.2.2.3 Quality Assurance/Quality Control Procedures
- 4.2.2.3.1 The Contractor shall strictly adhere to all specific QA/QC procedures prescribed in Exhibits D and E. Records documenting the use of the protocol shall be maintained in accordance with the document control procedures prescribed in Exhibit F, and shall be reported in accordance with Exhibits B and H.

- 4.2.2.3.2 The Contractor shall maintain a Quality Assurance Management Plan (QAP) with the objective of providing sound analytical chemical measurements. This program shall incorporate the QC procedures, any necessary corrective action, and all documentation required during data collection as well as the quality assessment measures performed by management to ensure acceptable data production.
- Additional QC shall be conducted in the form of the analysis of laboratory PE samples submitted to the laboratory by USEPA. Unacceptable results of all such QC or laboratory PE samples may be used as the basis for an equitable adjustment to reflect the reduced value of the data to USEPA or rejection of the data for specific analyte(s) within an SDG or the entire SDG. Also, unacceptable results may be used as the basis for contract action. "Compliant performance" is defined as that which yields correct analyte identification and concentration values as determined by USEPA, as well as meeting the contract requirements for analysis (Exhibit D); QA/QC (Exhibit E); data reporting and other deliverables (Exhibits B and H); and sample custody, sample documentation, and SOP documentation (Exhibit F).
- 4.2.3 Task III: Sample Reporting
- 4.2.3.1 USEPA has provided to the Contractor formats for the reporting of data (Exhibits B and H). The Contractor shall be responsible for completing and submitting analysis data sheets, computer-readable data on diskette (or via an alternate means of electronic transmission approved in advance by USEPA) in a format specified in this SOW and within the time specified in Exhibit B, Section 1.1.
- 4.2.3.2 Use of formats other than those designated by USEPA (see Exhibits B and H) will be deemed as noncompliant. Such data are unacceptable. Resubmission in the specified format at no additional cost to the Government shall be required.
- 4.2.3.3 Computer generated forms may be submitted in the hardcopy Sample Data Package(s) provided that the forms are in **exact USEPA format**. This means that the order of data elements is the same as on each USEPA required form, including form numbers and titles, page numbers, and header information.
- 4.2.3.4 The data reported by the Contractor on the hardcopy data forms and the associated computer-readable data submitted by the Contractor on diskette (or via an alternate means of electronic transmission, if approved in advance by USEPA) shall contain identical information. If discrepancies are found during Government inspection, the Contractor shall be required to resubmit either the hardcopy forms or the computer-readable data, or both sets of data, at no additional cost to USEPA.

EXHIBIT B

REPORTING AND DELIVERABLES REQUIREMENTS

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Exhibit B - REPORTING AND DELIVERABLES REQUIREMENTS

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1.0 CONTRACT REPORTS/DELIVERABLES DISTRIBUTION

1.1 Report Deliverable Schedule

The following table reiterates the contract reporting and deliverables requirements and specifies the distribution that is required for each deliverable. The turnaround times for Items B through E are 7, 14, or 21 days.

NOTE: Specific recipient names and addresses are subject to change during the term of the contract. The USEPA Office of Superfund Remediation and Technology Innovation (OSRTI) Analytical Services Branch (ASB) Inorganic Program Manager (ASB PM) will notify the Contractor in writing of such changes when they occur.

TABLE 1

				<u>D</u>)istri	butio	<u>n</u>
	Item	No. of Copies ^A	Delivery Schedule	OWS	Region	CLP PO ^D	QATS
Α.	Sample Traffic Reports/Chain of Custody Records	1	3 working days after receipt of last sample in Sample Delivery Group (SDG). ¹	Х			
B. ²	Sample Data Package	1	XX ^c days after Validated Time of Sample Receipt (VTSR) ¹ of last sample in SDG.	Х			
C. ²	Data in Computer- Readable Format	1	XX ^c days after VTSR of last sample in SDG.	Х	Х		
D. ²	Results of Intercomparison Study/PE Sample Analysis Study	1	XX ^c days after VTSR of last sample in SDG.	X			X
E. ^{2,3}	Complete SDG File (CSF) ^B	1	XX ^c days after VTSR of last sample in SDG.		Х		
F. ⁴	Preliminary Results	1	Within 72 hours after receipt of each sample at laboratory, if requested.	X	X		
G. ^{5,6}	Quarterly Verification of ICP-AES/ICP-MS Linear Ranges and ICP-AES Interelement Correction Factors	1	Quarterly: 15th day of January, April, July, and October.	Х		X	Х
	Annual Verification of Method Detection Limits (MDLs)	1	Annually: 15th day of January.	Х		Х	Х

TABLE 1 (Con't)

		N. C		Distribution			
Item		No. of Copies ^A	Delivery Schedule	SMO	Region	CLP PO ^D	QATS
H. ^{6,7}	Standard Operating Procedures (SOPs)	1	Revise within 30 days after contract award and receipt of USEPA comments. Submit within 7 days of receipt of written request to recipients as directed. (See Exhibit E, Section 6) Submit within 14 days of amended SOP(s) as directed in Exhibit E, Section 6.4.	A di:	As Din mende stribu P PO á	d SOP:	s .o
I. ^{6,7}	Quality Assurance Management Plan (QAP)	1	Revise within 30 days after contract award and receipt of USEPA comments. Submit within 7 days of receipt of written request to recipients as directed. (See Exhibit E, Section 5) Submit within 14 days of amended QAP as directed in Exhibit E, Section 5.3.	di	As Dir Amende stribu P PO a	ed QAF ited t	.0
J.	Electronic Instrument Data	Lot	Retain for 3 years after data submission. Submit within 7 days after receipt of written request by the USEPA Regional CLP PO. (See Exhibit E, Section 13)	7	As Dir	rected	l

Footnotes:

 $\,^{\text{A}}\text{The}$ number of copies specified is the number of copies required to be delivered to each recipient.

^BContractor-concurrent delivery to USEPA's designated recipient [e.g., Quality Assurance Technical Support (QATS)] may be required upon request by the USEPA OSRTI ASB Inorganic Program Manager (ASB PM). Retain for 365 days after data submission, and submit as directed within 7 days after receipt of written request by the USEPA ASB PM.

^CThe number of days associated with these elements will be provided in the associated laboratory contract document and will also be provided at the time of sample scheduling by the Sample Management Office (SMO) Contractor.

 $\,^{\text{D}}\!\text{The}$ CLP PO is the USEPA Regional Contract Laboratory Program (CLP) Project Officer (CLP PO) designated on the contract.

¹Validated Time of Sample Receipt (VTSR) is the date of sample receipt at the Contractor's facility, as recorded on the shipper's delivery receipt and sample Traffic Report/Chain of Custody Record. Sample Delivery Group (SDG) is a group of samples within a Case, received over a period of 7 days or less with the same laboratory turnaround and not exceeding 20 samples [excluding Performance Evaluation (PE) samples]. Data for all samples in the SDG are due concurrently. The date of delivery of the SDG or any samples within the SDG is the date that the last sample in the SDG is received. See Exhibit A for further description.

²DELIVERABLES ARE TO BE REPORTED TOTAL AND COMPLETE. Concurrent delivery is required. Delivery shall be made such that all designated recipients receive the item on the same calendar day. This includes resubmission of both the hardcopy and electronic deliverable. The date of delivery of the SDG, or any sample within the SDG, is the date all samples have been delivered. If the deliverables are due on a Saturday, Sunday, or Federal holiday, then they shall be delivered on the next business day. Deliverables received after this time will be considered late.

 3 Complete SDG File (CSF) will contain the original Sample Data Package plus all of the original documents described in Exhibit B, Section 2.6.

⁴If required at the time of sample scheduling, the Contractor shall provide Preliminary Results, consisting of all Form Is (see Exhibit B, Section 2.9). Facsimile or electronic transmittal is required as requested by the Region. Electronic transmittals shall be transmitted as WordPerfect, MS Word, PDF, or other USEPA-approved formats. The Contractor will be notified of the format, fax numbers, or email address(es) at the time of sample scheduling. Sample Traffic Reports/Chain of Custody Records and SDG Cover Sheets shall be submitted with the Preliminary Results. The Contractor shall document all communication in a telephone log.

Preliminary Results Delivery Schedule:

If a sample requiring Preliminary Results arrives before 5 p.m., the Preliminary Results are due within the required turnaround time. If a sample requiring Preliminary Results is received after 5 p.m., the Preliminary Results are due within the required turnaround time beginning at 8 a.m. the following day.

⁵Also required in each Sample Data Package.

⁶See Exhibit E for description. Time is cited in calendar days.

Exhibit B -- Section 1 Contract Reports/Deliverables Distribution (Con't)

Footnotes (Con't):

The Contractor shall deliver both hardcopy and electronic (i.e., diskette) copies of the Standard Operating Procedures (SOPs) and Quality Assurance Management Plan (QAP).

1.2 Distribution

The following addresses correspond to the "Distribution" column in Exhibit B, Section 1.1, Table 1.

SMO: USEPA Contract Laboratory Program (CLP)

Sample Management Office (SMO) 1 15000 Conference Center Drive Chantilly, VA 20151-3808

USEPA REGIONS: SMO will provide the Contractor with the list Region:

of addressees for data delivery for the 10 USEPA Regions. SMO will provide the Contractor with updated Regional

address/name lists as necessary throughout the period of the contract and identify other client recipients on a case-by-

case basis.

USEPA Regional CLP Project Officer (CLP PO):

SMO will provide the Contractor with the list of addresses for the USEPA Regional CLP POs. SMO will provide the Contractor with updated name/address lists as necessary

throughout the period of the contract.

QATS: USEPA Contract Laboratory Program (CLP)

Quality Assurance Technical Support (QATS) Laboratory²

2700 Chandler Avenue, Building C

Las Vegas, NV 89120 Attn: Data Audit Staff

In addition, the mailing and delivery addresses for the USEPA ASB Inorganic Program Manager (ASB PM) are:

Mailing Address: USEPA OSRTI Analytical Services Branch

Ariel Rios Building (5204G) 1200 Pennsylvania Avenue, N.W.

Washington, DC 20460

Attn: CLP Inorganic Program Manager

Delivery:

Fed-Ex/Overnight USEPA OSRTI Analytical Services Branch

1235 Jefferson Davis Highway Crystal Gateway I, 12th Floor

Arlington, VA 22202

Attn: CLP Inorganic Program Manager

¹The SMO is a Contractor-operated facility operating under the SMO contract awarded and administered by USEPA.

²The QATS laboratory is a Contractor-operated facility operating under the QATS contract awarded and administered by USEPA.

- 2.0 REPORTING REQUIREMENTS AND ORDER OF DATA DELIVERABLES
- 2.1 Introduction

The Contractor shall provide reports and other deliverables as specified in Exhibit B, Section 1.1. The required content and form of each deliverable is described in this exhibit. All reports and documentation shall be:

- Legible;
- Clearly labeled and completed in accordance with instructions in this exhibit;
- Arranged in the order specified in this section;
- · Paginated sequentially according to instructions in this exhibit; and
- Double-sided.

NOTE: Complete Sample Delivery Group (SDG) Files (CSFs) need not be double-sided. (The CSF is composed of original documents.) However, Sample Data Packages delivered to the USEPA Contract Laboratory Program (CLP) Sample Management Office (SMO) and the Region, [and USEPA designated recipients, e.g., Quality Assurance Technical Support (QATS), upon written request] must be double-sided.

- 2.1.1 The Contractor shall use EPA Case numbers, SDG numbers, and EPA sample numbers to identify samples received under this contract, both verbally and in reports and correspondence. The contract number shall be specified in all correspondence.
- 2.1.2 Section 4 of this exhibit contains the required Data Reporting Forms in Agency-specified format. Section 3 of this Exhibit contains instructions to the Contractor for properly completing all data reporting forms to provide USEPA with all required data. Data elements and field descriptors for reporting data in computer-readable format are contained in Exhibit H.
- 2.2 Resubmission of Data

If submitted documentation does not conform to the above criteria, the Contractor is required to resubmit such documentation with deficiency(ies) corrected within 4 business days, at no additional cost to USEPA.

- 2.2.1 Whenever the Contractor is required to submit or resubmit data as a result of an on-site laboratory evaluation, through the USEPA Regional CLP Project Officer (CLP PO) action, or through a Regional data reviewer's request, the data shall be clearly marked as "Additional Data" and shall be sent to both contractual data recipients (SMO and Region) and to USEPA's designated recipient (e.g., QATS) when a written request for the Sample Data Package has been made. A cover letter shall be included which describes what data is being delivered, to which USEPA Case(s) the data pertains, and who requested the data.
- 2.2.2 Whenever the Contractor is required to submit or resubmit data as a result of Contract Compliance Screening (CCS) review by SMO, the data shall be sent to the two contractual data recipients (SMO and Region) and to USEPA's designated recipient (e.g., QATS) when a written request for the Sample Data Package has been made. In all instances, the Contractor shall include a color-coded cover sheet (Laboratory

Response to Results of Contract Compliance Screening) provided by SMO. Electronic deliverables shall be submitted or resubmitted to SMO and the Region. Revised DC-1 and DC-2 forms shall be resubmitted to SMO and the Region.

Quality Assurance (QA) Management Plan and Standard Operating Procedures (SOPs)

The Contractor shall adhere to the requirements in Exhibits E and F.

2.4 Sample Traffic Reports/Chain of Custody Records

Each sample received by the Contractor will be labeled with an EPA sample number and will be accompanied by a Sample Traffic Report/Chain of Custody Record bearing the sample number and descriptive information regarding the sample. The current CLP Traffic Report is the "Inorganic Traffic Report & Chain of Custody Record". The CLP Traffic Report/Chain of Custody Record is one form divided into two sections: the Traffic Report section which consists of everything above the Chain of Custody Record section, and the bottom section which is the Chain of Custody Record. The Contractor shall complete the CLP Traffic Report/Chain of Custody Record (marked "Lab Copy for Return to SMO"), recording the date of sample receipt, verifying the number of samples, and signing the CLP Traffic Report/Chain of Custody Record.

Upon receipt, the Contractor shall sign for receipt of samples. The laboratory signature box is located at the bottom of the CLP Traffic Report/Chain of Custody Record in the Chain of Custody Record section. The laboratory sample custodian or designated recipient opening and verifying the contents of the cooler shall then verify receipt of all samples identified within the CLP Traffic Report section and sign and date the signature box located in the upper half of the CLP Traffic Report/Chain of Custody Record. If a non-CLP Traffic Report/Chain of Custody Record is submitted with the samples, for example a Regional Traffic Report/Chain of Custody Record, then the Contractor shall (1) sign and date receipt of the samples to maintain the chain-of-custody and (2) the sample custodian or designated recipient shall sign and date the Traffic Report/Chain of Custody Record to verify sample information.

The Contractor shall also enter the Sample Delivery Group (SDG) number, Case number, and the laboratory contract number on the CLP Traffic Report/Chain of Custody Record, in the appropriate boxes. The EPA sample number of the first sample received in the SDG is the SDG number. When several samples are received together in the first SDG shipment, the SDG number shall be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG. Under no circumstances should any SDG number be replicated within a Case. If necessary, select an alternative sample number for the SDG number. The SDG number is also reported on all data reporting forms (see Exhibit B, Section 3 - Form Instructions). If the laboratory is requested to transfer samples to another facility, the Contractor shall date and enter the name of the facility to where the samples will be transferred on the CLP Traffic Report/Chain of Custody Record.

2.4.1 The Contractor shall submit Traffic Reports/Chain of Custody Records in SDG sets (i.e., Traffic Reports/Chain of Custody Records for all samples in an SDG shall be clipped together), with an SDG Cover Sheet attached. The SDG Cover Sheet shall contain the following items:

- Laboratory name;
- Contract number;
- Sample analysis price (full sample price from the contract);
- Case number; and
- List of EPA sample numbers of all samples in the SDG, identifying the **first** and **last** samples received, and their Laboratory Receipt Dates (LRDs).

NOTE: When more than one sample is received in the first or last SDG shipment, the "first" sample received would be the sample with the lowest sample number (considering both alpha and numeric designations); the "last" sample received would be the sample with the highest sample number (considering both alpha and numeric designations).

- 2.4.2 EPA field sample numbers are six digits in length and continuous (without spaces or hyphens). If the Contractor receives sample numbers of any other length, the Contractor shall contact SMO immediately. The original Sample Traffic Report/Chain of Custody Record page marked "Lab Copy for Return to SMO", with laboratory receipt information and signed with original Contractor signature, shall be submitted for each sample in the SDG.
- 2.4.3 If samples are received at the laboratory with multi-sample Traffic Reports/Chain of Custody Records, all the samples on one multi-sample Traffic Report/Chain of Custody Record may not necessarily be in the same SDG. In this instance, the Contractor shall make the appropriate number of photocopies of the Traffic Report/Chain of Custody Record, and submit one copy with each SDG Cover Sheet.
- 2.5 Sample Data Package

The Sample Data Package shall include data for analysis of all samples in one SDG, including field and analytical samples, blanks, spikes, duplicates, and Laboratory Control Samples (LCSs). The Sample Data Package shall be complete before submission, and shall be consecutively paginated (starting with page number one and ending with the number of all pages in the package). The Sample Data Package shall include the following:

- 2.5.1 Cover Documentation
- 2.5.1.1 Cover Page for the inorganic analyses Data Package shall include: laboratory name; laboratory code; contract number; Case number; SDG number; Non-Routine Analytical Service (NRAS) number (if appropriate); EPA sample numbers in alphanumeric order showing EPA sample numbers cross-referenced with laboratory Sample ID numbers; and completion of the questions on use of background and interelement corrections for the samples.
- 2.5.1.1.1 The Cover Page shall contain the following statement, <u>verbatim</u>:
 "I certify that this Sample Data Package is in compliance with
 the terms and conditions of the contract, both technically and
 for completeness, for other than the conditions detailed above.
 Release of the data contained in this hardcopy Sample Data
 Package and in the computer-readable data submitted on diskette
 (or via an alternate means of electronic transmission, if
 approved in advance by USEPA) has been authorized by the
 Laboratory Manager or the Manager's designee, as verified by

the following signature." This statement shall be directly followed by the signature of the Laboratory Manager or designee with typed lines containing the signer's name and title, and the date of signature.

- SDG Narrative. This document shall be clearly labeled "SDG Narrative" and shall contain: laboratory name, Case number, SDG 2.5.1.2 number, contract number, and detailed documentation of any Quality Control (QC), sample, shipment, and/or analytical problems encountered in processing the samples reported in the Sample Data Package. The Contractor shall include any technical and administrative problems encountered and the resolution or corrective actions taken. This includes documenting the alternative technique used to determine cooler temperature if a temperature indicator bottle is not present in the cooler. The Contractor shall also provide, in the SDG Narrative, sufficient information, including equations or curves (at least one equation or curve per method), to allow the recalculation of sample results from raw instrument output. The Contractor shall also include a discussion of any flexibility Statement of Work (SOW) modification. This includes attaching a copy of the USEPA approved modification form to the SDG Narrative. Additionally the Contractor shall also identify and explain any differences which exist between the Form Is and supporting documentation provided in the data package and those previously provided as Preliminary Results.
- 2.5.1.3 Sample Log-In Sheet [Form DC-1]
- 2.5.1.4 Full Inorganics Complete SDG File (CSF) Inventory Sheet [Form DC-2]
- 2.5.1.5 Sample Traffic Reports/Chain of Custody Records
- 2.5.2 Sample Data

Sample data shall be submitted with the inorganic analysis data reporting forms for all samples in the SDG. Data should be arranged in increasing alphanumeric EPA sample number order, followed by the QC analyses data, quarterly and annual verification of method and instrument parameters forms, raw data, and copies of the digestion and distillation logs.

- 2.5.2.1 Inorganic Analysis Data Sheet [Form IA-IN and Form IB-IN].
 Tabulated analytical results of the requested analytes shall be included. The validation and release of these results is authorized by a specific signed statement on the Cover Page. In the event that the laboratory cannot verify all data reported for each sample, the Laboratory Manager shall provide a detailed description of the problems associated with the sample(s) in the SDG Narrative.
- 2.5.2.1.1 Appropriate concentration units shall be specified and entered on Forms IA-IN and IB-IN. The quantitative values shall be reported in units of micrograms per Liter (UG/L) for water samples and milligrams per kilogram (MG/KG) for solid samples. (No other units are acceptable.) Results for solid samples shall be reported on a dry weight basis. Analytical results shall be reported to two significant figures if the result value is less than 10 and to three significant figures if the value is greater than or equal to 10. Results for percent solids shall be reported to one decimal place. The preceding discussion concerning significant numbers applies to Forms IA-

IN, IB-IN, and IX-IN only. For other forms, follow the instructions specific to those forms as discussed in this exhibit.

- 2.5.2.2 Quality Control (QC) Data
- 2.5.2.2.1 The QC summary for inorganic analysis shall contain the forms listed below.

NOTE: If more than one form is necessary, duplicate forms must be arranged in chronological order.

- 2.5.2.2.1.1 Initial and Continuing Calibration Verification [Form IIA-IN]
- 2.5.2.2.1.2 CRQL Check Standard [Form IIB-IN]
- 2.5.2.2.1.3 Blanks [Form III-IN]
- 2.5.2.2.1.4 ICP-AES Interference Check Sample [Form IVA-IN]
- 2.5.2.2.1.5 ICP-MS Interference Check Sample [Form IVB-IN]
- 2.5.2.2.1.6 Matrix Spike Sample Recovery [Form VA-IN]
- 2.5.2.2.1.7 Post-Digestion Spike Sample Recovery [Form VB-IN]
- 2.5.2.2.1.8 Duplicates [Form VI-IN]
- 2.5.2.2.1.9 Laboratory Control Sample [Form VII-IN]
- 2.5.2.2.1.10 ICP-AES and ICP-MS Serial Dilutions [Form VIII-IN]
- 2.5.2.2.1.11 Method Detection Limits (Annually) [Form IX-IN]
- 2.5.2.2.1.12 ICP-AES Interelement Correction Factors (Quarterly) [Form XA-IN]
- 2.5.2.2.1.13 ICP-AES Interelement Correction Factors (Quarterly) [Form XB-IN]
- 2.5.2.2.1.14 ICP-AES and ICP-MS Linear Ranges (Quarterly) [Form XI-IN]
- 2.5.2.2.1.15 Preparation Log [Form XII-IN]
- 2.5.2.2.1.16 Analysis Run Log [Form XIII-IN]
- 2.5.2.2.1.17 ICP-MS Tune [Form XIV-IN]
- 2.5.2.2.1.18 ICP-MS Internal Standards Relative Intensity Summary [Form XV-IN]
- 2.5.2.3 Raw Data

For each reported value, the Contractor shall include in the Sample Data Package all raw data used to obtain that value. This applies to all required QA/QC measurements, instrument standardization, as well as all sample analysis results. This statement does not apply to the quarterly and annual verification of method and instrument parameters submitted as a part of each Sample Data Package. When analysis of the ICP-AES or ICP-MS target analytes listed in Exhibit C of this SOW (or any subset or additional analytes) is requested, the raw data shall include, for

all samples, not only the results for the requested analyte(s), but also those for all the interferents (Exhibit D/ICP-AES, Table 1, or Exhibit D/ICP-MS, Section 7.2.4.4.1, as appropriate). The raw data shall also contain the results of any other analyte(s) which have been determined to interfere with the requested analytes(s).

- 2.5.2.3.1 Raw data shall contain all instrument readouts and data pertinent to the reconstruction of the analysis and results (e.g., Batch Sheets) used for the sample results. Each exposure or instrumental reading shall be provided, including those readouts that may fall below the Method Detection Limit (MDL). Raw data shall not be corrected for dilutions or volume adjustments. All Atomic Absorption (AA), Inductively Coupled Plasma - Atomic Emission Spectrometer (ICP-AES), and Inductively Coupled Plasma - Mass Spectrometer (ICP-MS) instruments shall provide a legible hardcopy of the direct real-time instrument readout (i.e., strip charts, printer tapes, etc.) or a printout of the unedited instrument data output file. A photocopy of the instrument's direct sequential readout shall be included. A hardcopy of the instrument's direct readout shall be included for cyanide if the instrumentation has the capability.
- 2.5.2.3.2 The order of raw data in the Sample Data Package for inorganic analyses shall be: ICP-AES, Graphite Furnace Atomic Absorption (GFAA), ICP-MS, Mercury, and Cyanide. All raw data shall include concentration units for ICP, and absorbances or concentration units for Mercury and Cyanide.
- 2.5.2.3.3 Corrections to the laboratory data reporting forms and raw data shall be made by drawing single lines through the errors and entering the correct information. Information shall not be obliterated or rendered unreadable. Corrections and additions to information shall be signed (or initialed) and dated.
- 2.5.2.3.4 Raw data shall be labeled with EPA sample numbers and appropriate codes, shown in Exhibit B, Table 2 Codes for Labeling Data, following, to unequivocally identify:
 - Calibration standards, including source and preparation date. Standard preparation logbooks can be submitted if they contain this information;
 - Initial and Continuing Calibration Blanks (ICBs/CCBs) and Preparation Blanks (PBs);
 - Initial and Continuing Calibration Verification (ICV/CCV) standards, Interference Check Samples (ICSs), serial dilution samples, Contract Required Quantitation Limit (CRQL) Check Standard (CRI), LCS, and post digestion spike;
 - Diluted and undiluted samples (by EPA sample number) and all weights, dilutions, and volumes used to obtain the reported values (if the volumes, weights, and dilutions are consistent for all samples in a given SDG, a general statement outlining these parameters is sufficient);
 - Duplicates;
 - Spikes (indicating standard solutions used, final spike concentrations, and volumes involved). If spike information (source, concentration, volume) is consistent

for a given SDG, a general statement outlining these parameters is sufficient;

- Instrument used, any instrument adjustments, data corrections or other apparent anomalies on the measurement record, including all data voided or data not used to obtain reported values and a brief written explanation; and
- Time and date of each analysis. Instrument run logs can also be submitted if they contain time and date of analysis. If the instrument does not automatically provide times of analysis, these shall be manually entered on all raw data (e.g., ICV/CCV, blanks, and the CRQL Check Standard).

Table 2	
Codes for Labeling	Data ^{1,2}
Sample	XXXXXX
Sample Not Part of the SDG	ZZZZZZ
Duplicate	XXXXXXD
Matrix Spike	XXXXXXS
Serial Dilution	XXXXXXL
Analytical Spike/Post	XXXXXXA
Digestion/Distillation Spike	
Instrument Calibration Standards:	
ICP	S or SO for blank standard
Atomic Absorption and Cyanide	S0, S10,etc.
Initial Calibration Verification	ICV
Initial Calibration Blank	ICB
Continuing Calibration Verification	CCV
Continuing Calibration Blank	CCB
Interference Check Samples:	
Solution A	ICSA
Solution AB	ICSAB
CRQL Check Standard	CRI
Laboratory Control Samples:	
Aqueous (Water)	LCSW
Solid (Soil/Sediment)	LCSS
Preparation Blank (Water)	PBW
Preparation Blank (Soil)	PBS
Linear Range Analysis Standard	LRS
Baseline Correction	BASELINE
Reslope	RESLOPE
Cyanide Mid-Range Standard	MIDRANGE
ICP-MS Tune Check	TUNE

 1 The numeric suffix that follows the "S" suffix for the standards indicates the true value of the concentration of the standard in ug/L.

 $^2\mbox{ICP-AES}$ and ICP-MS calibration standards usually consist of several analytes at different concentrations. Therefore, no numeric suffix can follow the ICP calibration standards unless all the analytes in the standard are prepared at the same concentrations. For instance, the blank for ICP shall be formatted "SO".

- 2.5.2.4 Digestion and Distillation Logs. The following logs shall be submitted as appropriate for each preparation procedure: digestion logs for ICP-AES, ICP-MS, mercury preparations, and cyanide. These logs shall include: (1) date; (2) sample weights and volumes, with initial sample weight/volume and final volume clearly indicated; (3) sufficient information to unequivocally identify which QC samples (i.e., LCS, PB) correspond to each batch digested; (4) comments describing any significant sample changes or reactions which occur during preparation shall be entered in the log and noted in the SDG Narrative; (5) indication of pH less than 2 or greater than 12, as applicable; and (6) identification of the sample preparer(s) [signature(s)].
- 2.6 Complete SDG File (CSF)

As specified in the Delivery Schedule, one CSF (including the original Sample Data Package) shall be delivered to the Region concurrently with the delivery of a copy of the Sample Data Package to SMO. Delivery to USEPA's designated recipient (e.g., QATS) is only required upon written request.

- 2.6.1 The CSF shall contain all original documents where possible. No photocopies of original documents shall be placed in the CSF unless the original data was initially written in a bound notebook, maintained by the Contractor, or the originals were previously submitted to USEPA with another Case/SDG in accordance with the requirements described in Exhibit F. The CSF shall contain all original documents and be numbered according to the specifications in Exhibit B, Sections 3 and 4, and Form DC-2.
- 2.6.2 The CSF shall consist of the following original documents in addition to the documents in the Sample Data Package.

NOTE: All Case-related documentation may be used or admitted as evidence in subsequent legal proceedings. Any other Case-specific documents generated after the CSF is sent to USEPA, as well as copies that are altered in any fashion, are also deliverables to USEPA. Send the original to the Region and a copy to SMO. Send to USEPA's designated recipient (e.g., QATS) only upon written request.

- 2.6.2.1 Original Sample Data Package
- 2.6.2.2 A completed and signed Full Inorganics Complete SDG File (CSF) Inventory Sheet [Form DC-2]
- - USEPA Sample Traffic Reports/Chain of Custody Records
 - Airbills (if an airbill is not received, include a hardcopy receipt requested from the shipping company or a printout of the shipping company's electronic tracking information); and
 - Sample Tags (if present) sealed in plastic bags.

- 2.6.2.4 All original receiving documents, including, but not limited to, the following documents:
 - Form DC-1;
 - Other receiving forms or copies of receiving logbooks; and
 - SDG Cover Sheet.
- 2.6.2.5 All original laboratory records of sample transfer, preparation, and analysis, including, but not limited to, the following documents:
 - Original preparation and analysis forms or copies of preparation and analysis logbook pages; and
 - Internal sample and sample digestate and distillate transfer Chain of Custody Records.
- 2.6.2.6 All other original SDG-specific documents in the possession of the laboratory, including, but not limited to, the following documents:
 - Telephone contact logs;
 - Copies of personal logbook pages;
 - All handwritten SDG-specific notes; and
 - Any other SDG-specific documents not covered by the above.
- 2.6.3 If the Contractor does submit SDG-specific documents to USEPA after submission of the CSF, the documents shall be numbered as an addendum to the CSF and a revised Form DC-2 shall be submitted; or the documents shall be numbered as a new CSF and a new Form DC-2 shall be submitted to the Region only.
- 2.6.4 The Contractor shall retain a legible electronic (PDF) or hard copy of the CSF for 365 days after submission of the reconciled data package. After this time, the Contractor may dispose of the package.
- 2.7 Data in Computer-Readable Format

The Contractor shall provide a computer-readable copy for all samples in the SDG, as specified in Exhibit H, and delivered as specified in Exhibit B, Section 1.1. Computer-readable data deliverables shall be submitted on DOS formatted 3.5-inch high density 1.44 MB diskette(s) (or via an alternate means of electronic transmission, if approved in advance by USEPA).

- 2.7.1 When submitted, diskette(s) shall be packaged and shipped in such a manner that the diskette(s) cannot be bent or folded and will not be exposed to extreme heat/cold or any type of electromagnetic radiation. The diskette(s) shall be included in the same shipment as the hardcopy data, and, at a minimum, be enclosed in a diskette mailer.
- 2.7.2 The data shall be recorded in the file format and adhere to the file, record, and field specifications listed in Exhibit H, "Data Dictionary and Format for Data Deliverables in Computer-Readable Format".

2.8 Results of the Intercomparison and Performance Evaluation (PE) Sample Analyses

Tabulation of analytical results for intercomparison/PE sample analyses includes all requirements specified in Exhibit B, Sections 2.5 and 2.7.

2.9 Preliminary Results

The Form Is data results (including all appropriate qualifiers and flags) shall be submitted for all samples in one SDG of a Case. Sample analysis shall follow all requirements stipulated in Exhibit D. The Contractor shall clearly identify the Preliminary Results by labeling each Form I as "Preliminary Results" under the form title (e.g., under Inorganic Analysis Data Sheet). The Contractor shall also include a disclaimer in the "Comments" field on all Form Is stating that the "Data results contained on this Form I are for scanning purposes only, and may not have been validated for CLP criteria." Sample Traffic Reports/Chain of Custody Records and SDG Cover Sheets shall be submitted with the Preliminary Results.

- 2.9.1 The Contractor shall submit the Cover Page following the specifications in Exhibit B, Sections 2.5.1 and 3.4.1. The Cover Page shall be clearly labeled to indicate that the data being reported are Preliminary Results. The Cover Page shall contain the following statement, verbatim: "I certify that these Preliminary Results are in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature."

 This statement shall be directly followed by the signature of the Laboratory Manager or designee with typed lines containing the signer's name and title, and the date of signature.
- 2.10 Quarterly Verification of Linear Ranges and Interelement Correction Factors and Annual Verification of MDLs

The Contractor shall perform and report quarterly verification of instrument linear range and annual verification of MDLs by the methods specified in Exhibit D for each instrument used under this contract. The Contractor shall also perform and report quarterly ICP-AES interelement correction factors (including method of determination), wavelengths used, and integration times. Forms reporting results for quarterly and annual verification of method and instrument parameters for the current quarter and year shall be submitted in each Sample Data Package, using Inorganic Forms IX, XA, XB, and XI. Submission of the quarterly and annual verification of method and instrument parameters shall include the raw data used to determine the values reported.

2.11 Electronic Instrument Data

The Contractor shall adhere to the requirements in Exhibit E.

2.12 Corrective Action Procedures

If the Contractor fails to adhere to the requirements detailed in this SOW, the Contractor will be in noncompliance with the contract and may be subjected to sanctions as described in the contract.

- 3.0 FORM INSTRUCTIONS
- 3.1 Introduction

This section contains specific instructions for the completion of all required Inorganic Data Reporting Forms.

3.2 General Information

Values shall be reported on the hardcopy forms according to the respective form instructions in this section. Each form submitted shall be filled out completely for all analytes before proceeding to the next form of the same type. Do not submit multiple forms if the information on those forms can be submitted on one form.

3.2.1 The data reporting forms discussed in Exhibit B, Section 3.4, and presented in Exhibit B, Section 4.0, have been designed in conjunction with the computer-readable data formats specified in Exhibit H, "Data Dictionary and Format for Data Deliverables in Computer-Readable Format". The specific length of each variable for computer-readable data transmission purposes is given in Exhibit H. Information entered on these forms shall **not** exceed the size of the field given on the form, including such laboratory-generated items as "Lab Name" and "Lab Sample ID".

NOTE: On the hardcopy forms, the space provided for entries is greater in some instances than the length prescribed for the variable as written to the electronic deliverable (see Exhibit H). Greater space is provided on the hardcopy forms for the sake of visual clarity.

- 3.2.2 All characters which appear on the data reporting forms presented in the contract shall be reproduced by the Contractor when submitting data, and the format of the forms submitted shall be identical to that shown in the contract. No information may be added, deleted, or moved from its specified position without prior written approval of the USEPA Regional Contract Laboratory Program Project Officer (CLP PO) or the USEPA Office of Superfund Remediation and Technology Innovation (OSRTI) Analytical Services Branch (ASB) Inorganic Program Manager (ASB PM). The names of various fields and analytes (i.e., "Lab Code", "Aluminum") shall appear as they do on the forms in the contract, including the options specified in the form (i.e., "Matrix (soil/water):" shall appear, not just "Matrix").
- Alphabetic entries made onto the forms by the Contractor shall be in ALL UPPERCASE letters (i.e., "LOW", not "Low" or "low"). If an entry does not fill the entire blank space provided on the form, null characters shall be used to remove the remaining underscores that comprise the blank line (see Exhibit H for additional instructions). However, do not remove the underscores or vertical bar characters that delineate "boxes" on the forms.
- 3.3 Header Information

Six pieces of information are common to the header sections of each data reporting form. These are: Laboratory Name, Contract, Laboratory Code, Case number, Non-Routine Analytical Services (NRAS) number, and Sample Delivery Group (SDG) number. Except as noted for NRAS number, this information shall be entered on every form and shall match on all forms.

3.3.1 Laboratory Name. The "Lab Name" shall be the name chosen by the Contractor to identify the laboratory. It may not exceed 25 characters.

ILM05.3

- 3.3.2 Contract. The "Contract" is the number of the USEPA contract under which the analyses were performed.
- 3.3.3 Laboratory Code. The "Lab Code" is an alphabetic abbreviation of up to six characters, <u>assigned by USEPA</u>, to identify the laboratory and aid in data processing. This laboratory code will be assigned by USEPA at the time a contract is awarded. The laboratory code <u>shall not</u> be modified by the Contractor, except at the direction of USEPA. If a change of name or ownership occurs at the laboratory, the laboratory code will remain the same until the Contractor is directed by USEPA to use another laboratory code.
- 3.3.4 Case Number. The "Case No." is the SMO-assigned Case number (to five characters) associated with the sample, and reported on the Traffic Report/Chain of Custody Record.
- 3.3.5 NRAS Number. The "NRAS No." is the USEPA assigned number for analyses performed under Non-Routine Analytical Services (NRAS). If samples are to be analyzed under NRAS only, and reported on these forms, then enter the NRAS number and leave the Case number blank. If samples are analyzed according to the Routine Analytical Services (RAS) protocol and have additional NRAS requirements, list both the Case number and NRAS number on all forms. If the analyses have no NRAS requirements, leave the "NRAS No." field blank.
- 3.3.6 SDG Number. The "SDG No." is the Sample Delivery Group (SDG) number. The SDG number is the EPA sample number of the first sample received in the SDG, except when this would cause duplication. When several samples are received together in the first SDG shipment, the SDG number shall be the lowest sample number (considering both alpha and numeric designations) in the first group of samples received under the SDG. If fractions of the same field samples are scheduled under different turnaround times, thus creating separate SDGs containing the same sample numbers, a different sample number shall be utilized in the assignment of the SDG number for each SDG. If a situation arises where there are an insufficient number of samples for assignment of SDG numbers, the contractor shall contact SMO for the assignment of a SDG number.
- 3.3.7 Sample Number. The "EPA Sample No." appears either in the header information of the form or as the left column of a table summarizing data from a number of samples. When an EPA sample number is entered in the triple-spaced box in the upper right-hand corner of a form, it shall be centered on the middle line of the three lines that form the box.
- 3.3.7.1 All samples, matrix spikes, post digestion/distillation spikes, duplicates, and serial dilutions shall be identified with an EPA sample number. For samples, an EPA sample number is the unique identifying number given in the Traffic Report/Chain of Custody Record that accompanied that sample. In order to facilitate data assessment, the sample suffixes listed in Exhibit B, Table 2 Codes for Labeling Data, must be used.

- 3.3.8 Other Common Fields. Other pieces of information are common to many of the data reporting forms. These include Matrix and Level.
 - For "Matrix", enter "SOIL" for soil/sediment samples and "WATER" for water samples.

NOTE: The matrix must be spelled out. Abbreviations such as "S" or "W" shall ${f not}$ be used.

- For "Level", enter the determination of concentration level. Enter as "LOW" or "MED", not "L" or "M".
- 3.3.9 Rounding Rule. For rounding off numbers to the appropriate level of precision, observe the following common rules. If the figure following those to be retained is greater than or equal to 5, the absolute value of the result is to be rounded up; otherwise the absolute value of the result is rounded down. For example, -0.4365 rounds to -0.437 and -2.3564 rounds to -2.356. Also see "Rounding Rules" in Exhibit G.
- 3.3.9.1 Before evaluating a number for being in control or out of control of a certain limit [other than the Contract Required Quantitation Limit (CRQL)], the number evaluated shall be rounded using the above rounding rules to the significance reported for that limit. For example, the control limit for an Initial Calibration Verification is plus or minus 10% of the true value. Then a calculated percent recovery of 110.46 shall be reported on Form IIA-IN as 110, which is within the control limits of 90-110. On the other hand, a calculated percent recovery of 110.50 shall be reported on Form IIA-IN as 111, which is not within the 90-110 percent control limits.

NOTE: All results shall be transcribed to Inorganic Forms IIA-IN through XV-IN from the raw data to the specified number of decimal places that are described in Exhibits B and H. The raw data result is to be rounded only when the number of figures in the raw data result exceeds the maximum number of figures specified for that result entry for that form. If there are not enough figures in the raw data result to enter in the specified space for that result, then zeros shall be used for decimal places to the specified number of reporting decimals for that result for a specific form. The following examples are provided:

Raw Data Result	Specified Format	Correct Entry on Form
95.99653	5.4 (to four decimal places)	95.9965
95.99653	5.3 (to three decimal places	95.997
95.99653	5.2 (to two decimal places)	96.00
95.996	5.4 (to four decimal places)	95.9960
95.9	5.4 (to four decimal places)	95.9000

- 3.4 Inorganic Forms
- 3.4.1 Cover Page [COVER PAGE]
- 3.4.1.1 Purpose. This form is used to list all samples analyzed within an SDG and provide certain analytical information and general comments. It is also the document that is signed by the Laboratory Manager to authorize and release all data and deliverables associated with the SDG.

- 3.4.1.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.
- 3.4.1.2.1 For samples analyzed using this Statement of Work (SOW), enter "ILM05.3" for the SOW Number.
- 3.4.1.2.2 Enter an EPA sample number including spikes and duplicates (to seven spaces) of every sample analyzed within the SDG. Spikes shall contain an "S" suffix and duplicates a "D" suffix. These sample numbers shall be listed on the form in ascending alphanumeric order. Thus, if MAB123 is the lowest (considering both alpha and numeric characters) EPA sample number within the SDG, it would be entered in the first EPA sample number field. Samples would be listed below it, in ascending sequence MAB124, MAB125, MAC111, MA1111, MA1111D, etc.
- 3.4.1.2.3 A maximum of 20 field sample numbers (excluding PE samples) can be entered on this form. Submit additional Cover Pages, as appropriate, if the total number of samples, duplicates, and spikes in the SDG is greater than 22.
- 3.4.1.2.4 A Laboratory Sample ID (to ten spaces) may be entered for each EPA sample number. If a Laboratory Sample ID is entered, it shall be entered identically (for each EPA sample number) on all associated data.
- 3.4.1.2.5 Enter "YES" or "NO" in answer to each of the two questions concerning Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) and Inductively Coupled Plasma Mass Spectrometry (ICP-MS) corrections. Each question shall be explicitly answered with a "YES" or a "NO". The third question shall be answered with a "YES" or "NO" if the answer to the second question is "YES". It shall be left blank if the answer to the second question is "NO".
- 3.4.1.2.6 Under "Comments", enter any statements relevant to the analyses performed under the SDG as a whole.
- 3.4.1.2.7 Each Cover Page shall be signed and dated, in original, by the Laboratory Manager or the Manager's designee to authorize the release and verify the contents of all data and deliverables associated with an SDG.
- 3.4.2 Inorganic Analysis Data Sheet [Forms IA-IN and IB-IN]
- 3.4.2.1 Purpose. These forms are used to tabulate and report sample analysis results for inorganic target analytes (see Exhibit C).
- 3.4.2.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.
- 3.4.2.2.1 "Date Received" is the date (formatted MM/DD/YYYY) of sample receipt at the laboratory, as recorded on the Traffic Report/Chain of Custody Record [i.e., the Validated Time of Sample Receipt (VTSR)].
- 3.4.2.2.2 "% Solids" is the percent of solids on a weight-by-weight basis in the sample which is determined by drying the sample as specified in Exhibit D Introduction to Analytical Methods, Section 1.6. Report percent solids to one decimal place (i.e.,

5.3%). If the percent solids is not required because the sample is fully aqueous, or is less than 1% solid, then enter "0.0".

- 3.4.2.2.3 Enter the appropriate concentration units (UG/L for water or MG/KG dry weight for soil). Entering "MG/KG" means "mg/kg dry weight" on this form.
- 3.4.2.2.4 Under the column labeled "Concentration", enter for each analyte, the value of the result [if the concentration is greater than or equal to the Method Detection Limit (MDL)] corrected for any dilutions; or, enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions, if the concentration is less than the MDL. The concentration result shall be reported to two significant figures if the result is less than 10 or three significant figures if the value is greater than or equal to 10.
- 3.4.2.2.5 Under the columns labeled "C", "Q", and "M", enter result qualifiers as identified below. If additional qualifiers are used, their explicit definitions shall be included on the Cover Page in the "Comments" section.

Forms IA-IN and IB-IN include fields for three types of result qualifiers. These qualifiers shall be completed as follows:

3.4.2.2.5.1 C (Concentration) Qualifier. Enter "J" if the reported value was obtained from a reading that was less than the CRQL but greater than or equal to the MDL. If the reading was less than the MDL, a "U" shall be entered.

The MDL obtained for a given preparation method, analysis method, and instrument shall be used for qualification of the results for samples associated with that preparation method, analysis method, and instrument. Serial dilution and post-digestion spike results shall be qualified using the MDL and CRQL values utilized for the corresponding field sample.

All three values (i.e., the instrument reading, CRQL, and MDL) shall be converted to the same units prior to determining the appropriate C (Concentration) Qualifier.

NOTE: The water CRQL (in ug/L) and the MDL obtained from direct analysis (Preparation Method "NP1") for a given analysis method and instrument shall be used to qualify the results of instrument QC standards that are not taken through a preparation procedure (e.g., ICB, CCB, and CRI for ICP-AES].

3.4.2.2.5.2 Q Qualifier. Specified entries and their meanings are as follows:

E: The reported value is estimated due to the presence of interference. An explanatory note shall be included under "Comments" on the Cover Page (if the problem applies to all samples), or on the specific Form IA-IN or Form IB-IN (if it is an isolated problem).

- N: Spiked sample recovery not within control limits.
- *: Duplicate analysis not within control limits.
- D: The reported value is from a dilution.
- 3.4.2.2.5.3 M (Analysis Method) Qualifier. Specified entries and their meanings are as follows:
 - P: ICP-AES
 - MS: ICP-MS
 - CV: Manual Cold Vapor Atomic Absorption (AA)
 - AV: Automated Cold Vapor AA
 - AS: Semi-Automated Spectrophotometric
 - C: Manual Spectrophotometric
 - "": Where no data have been entered
 - NR: If the analyte is not required to be analyzed
- 3.4.2.2.6 A brief physical description of the sample, both before and after digestion, shall be reported in the fields for color (before and after), clarity (before and after), texture, and artifacts. For water samples, report color and clarity. For soil samples, report color, texture, and artifacts. The following descriptive terms are recommended:
 - Color red, blue, yellow, green, orange, violet, white, colorless, brown, grey, and black;
 - Clarity clear, cloudy, and opaque; and
 - Texture fine (powdery), medium (sand), and coarse (large crystals or rocks).

If artifacts are present, enter "YES" in the artifacts field and describe the artifacts in the "Comments" field. If artifacts are not present, leave this field blank. Note any significant changes that occur during sample preparation (i.e., emulsion formation) in the "Comments" field. Enter any sample-specific comments concerning the analyte results in the "Comments" field. Also document raw instrument results that are less than minus two times the CRQL (-2xCRQL) in the "Comments" field and in the Sample Delivery Group (SDG) Narrative.

- 3.4.2.2.7 If more than two additional analytes were requested, submit Form IB-IN as appropriate.
- 3.4.3 Initial (ICV) and Continuing Calibration Verification (CCV) [Form IIA-IN]
- 3.4.3.1 Purpose. This form is used to report analyte recoveries from calibration verification solutions.
- 3.4.3.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.
- 3.4.3.2.1 Enter the ICV Source (12 characters maximum) and the CCV Source (12 characters maximum). Enter sufficient information in the available 12 spaces to identify the manufacturer and the solution used.

Use additional Form(s) IIA-IN if more calibration verification sources were used.

- 3.4.3.2.2 Under "Initial Calibration Verification True", enter the value [in micrograms per Liter (ug/L), to one decimal place] of the concentration of each analyte in the ICV Solution.
- 3.4.3.2.3 Under "Initial Calibration Verification Found", enter the most recent value (in ug/L, to two decimal places), of the concentration of each analyte measured in the ICV Solution.
- 3.4.3.2.4 Under "Initial Calibration Verification %R", enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:
 - EQ. 1 ICV Percent Recovery

$$R = \frac{\text{Found}(I \text{ CV})}{\text{True}(ICV)} \times 100$$

WHERE, "True(ICV)" is the true concentration of the analyte in the ICV Solution and "Found(ICV)" is the found concentration of the analyte in the ICV Solution.

The values used in EQ. 1 for "True(ICV)" and "Found(ICV)" shall be exactly those reported on this form.

- 3.4.3.2.5 Under "Continuing Calibration Verification True", enter the value (in ug/L, to one decimal place) of the concentration of each analyte in the CCV Solution.
- 3.4.3.2.6 Under "Continuing Calibration Verification Found", enter the value (in ug/L, to two decimal places) of the concentration of each analyte measured in the CCV Solution.

NOTE: The form contains two "Continuing Calibration Verification Found" columns. The column to the left shall contain values for the first CCV, and the column to the right shall contain values for the second CCV.

- 3.4.3.2.7 If more than one Form IIA-IN is required to report multiple CCVs, then the column to the left on the second form shall contain values for the third CCV, the column to the right shall contain values for the fourth CCV, and so on.
- 3.4.3.2.8 Under "Continuing Calibration Verification %R", enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:
 - EQ. 2 CCV Percent Recovery

$$%R = \frac{Found(CCV)}{True(CCV)} \times 100$$

WHERE, "True(CCV)" is the true concentration of each analyte, and "Found(CCV)" is the found concentration of the analyte in the CCV Solution.

The values used in EQ. 2 for "True(CCV)" and "Found(CCV)" shall be exactly those reported on this form.

Exhibit B -- Section 3 Form Instructions Form IIB-IN

NOTE: The form contains two "Continuing Calibration Verification %R" columns. Entries to these columns shall follow the sequence detailed above for entries to the "Continuing Calibration Verification Found" columns.

- 3.4.3.2.9 Under "M", enter the method used or "NR", as explained in Exhibit B, Section 3.4.2.2.5.3.
- 3.4.3.2.10 If more than one wavelength/mass is used to analyze an analyte, submit additional Form(s) IIA-IN as appropriate.
- 3.4.3.2.11 The order of reporting ICVs and CCVs for each analyte shall follow the chronological order in which the standards were run. Start with the first Form IIA-IN and move from the left to the right, continuing to the following Form IIA-INs as appropriate. For instance, the first ICV for all analytes shall be reported on the first Form IIA-IN. In a run where three CCVs were analyzed, the first CCV shall be reported in the left CCV column on the first Form IIA-IN and the second CCV shall be reported in the right column of the same form. The third CCV shall be reported in the left CCV column of the second Form IIA-IN. On the second Form IIA-IN, the ICV column and the right CCV column shall be left empty in this example. In the previous example, if a second run for an analyte was needed, the ICV of that run shall be reported on a third Form IIA-IN and the CCVs follow in the same fashion as explained before. In the case where two wavelengths are used for an analyte, all ICV and CCV results of one wavelength from all runs shall be reported before proceeding to report the results of the second wavelength used.
- 3.4.4 CRQL Check Standard [Form IIB-IN]
- 3.4.4.1 Purpose. This form is used to report analyte recoveries from analyses of the CRQL Check Standards (CRIs).
- 3.4.4.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.
- 3.4.4.2.1 Enter the CRQL Check Standard Source (12 characters maximum) as explained in Exhibit B, Section 3.4.3.2.1.
- 3.4.4.2.2 Under "CRQL Check Standard True", enter the value (in ug/L, to one decimal place) of the concentration of each analyte in the CRQL Check Standard that was analyzed for analytical samples associated with the SDG.
- 3.4.4.2.3 Under "CRQL Check Standard Initial Found", enter the result (in ug/L, to two decimal places) measured in the CRQL Check Standard analyzed at the beginning of the run. For each analyte, enter the value of the result (if the concentration is greater than or equal to the MDL); or enter the CRQL of the analyte if the concentration is less than the MDL. If applicable, enter the concentration qualifier "J" or "U" after the concentration (e.g., 1.96J for Lead), as specified in Exhibit B, Section 3.4.2.2.5.1.
- 3.4.4.2.4 Under "CRQL Check Standard Initial R", enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:

EQ. 3 CRQL Check Standard Initial Percent Recovery

$\label{eq:R} \$R = \frac{\text{CRQL Check Standard Initial Found}}{\text{CRQL Check Standard True}} x 100$

- 3.4.4.2.5 Under "CRQL Check Standard Final Found", enter the results (in ug/L, to two decimal places) measured in the CRQL Check Standard(s) analyzed after the beginning of the run. For each analyte, enter the value of the result (if the concentration is greater than or equal to the MDL); or enter the CRQL of the analyte if the concentration is less than the MDL. If applicable, enter the concentration qualifier "J" or "U" after the concentration (e.g., 1.96J for Lead), as specified in Exhibit B, Section 3.4.2.2.5.1.
- 3.4.4.2.6 Under "CRQL Check Standard Final %R", enter the value (to the nearest whole number) of the percent recovery computed according to the following equation:
 - EQ. 4 CRQL Check Standard Final Percent Recovery

$$\label{eq:R} \text{%R} = \frac{\text{CRQL Check Standard Final Found}}{\text{CRQL Check Standard True}} \times 100$$

3.4.4.2.7 All percent recovery values reported in EQs. 3 and 4 shall be calculated using the exact true and found values reported on this form. A value of zero shall be used in calculations if the analyte value is less than the MDL.

NOTE: For every initial solution reported there must be a final one. However, the opposite is <u>not</u> true. If a CRQL Check Standard was required to be analyzed in the middle of a run, it shall be reported in the "Final Found" section of this form.

- 3.4.4.2.8 If more CRI analyses were required or analyses were performed using more than one wavelength per analyte, submit additional Form(s) IIB-IN as appropriate.
- 3.4.4.2.9 The order of reporting CRIs for each analyte shall follow the chronological order in which the standards were run starting with the first Form IIB-IN and continuing to the following Forms IIB-IN as appropriate. When multiple wavelengths are used for one analyte, all the results of one wavelength shall be reported before proceeding to the next wavelength.
- 3.4.5 Blanks [Form III-IN]
- 3.4.5.1 Purpose. This form is used to report analyte concentrations found in the Initial Calibration Blank (ICB), Continuing Calibration Blanks (CCB), and the Preparation Blank (PB).
- 3.4.5.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.
- 3.4.5.2.1 Enter "SOIL" or "WATER" as appropriate as the matrix of the PB.
 No abbreviations or other matrix descriptors may be used.

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Form III-IN (Con't)

- 3.4.5.2.2 According to the matrix specified for the PB, enter the PB concentration units as "UG/L" for water or "MG/KG" for soil.
- 3.4.5.2.3 Under "Initial Calibration Blank", enter the concentration (in ug/L, to three decimal places) of each analyte in the most recent ICB, as described in Exhibit B, Section 3.4.5.2.8, below.
- 3.4.5.2.4 For each calibration blank associated with a given method and instrument, enter "J" under the "C" qualifier field on Form III-IN if the absolute value of the analyte concentration is less than the CRQL for water but greater than or equal to the MDL that was obtained from direct analysis (Preparation Method "NP1") using that method and instrument.

For prepared calibration blanks (e.g., mercury), the CRQL for water and the MDL for the preparation method, analysis, and instrument shall be used.

Enter "U" if the absolute value of the analyte in the blank is less than the MDL obtained from direct analysis or the preparation method.

- 3.4.5.2.5 Under "Continuing Calibration Blank 1", enter the concentration (in ug/L, to three decimal places) of each analyte detected in the first required CCB analyzed after the ICB, as described in Exhibit B, Section 3.4.5.2.8, below. Enter any appropriate qualifier, as explained for the "Initial Calibration Blank", to the "C" qualifier column immediately following the "Continuing Calibration Blank 1" column.
- 3.4.5.2.6 If up to three CCBs were analyzed, complete the columns labeled "2" and "3" in accordance with the instructions for the "Continuing Calibration Blank 1" column. If more than three CCBs were analyzed, then complete additional Form(s) III-IN as appropriate.
- 3.4.5.2.7 Under "Preparation Blank", enter the concentration in ug/L (to three decimal places) for a water blank, or mg/kg (to three decimal places) for a soil blank, of each analyte in the PB, as described in Exhibit B, Section 3.4.5.2.8, below. Evaluate the absolute value of the analyte concentration to determine the appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, and enter the qualifier in the "C" column immediately following the "Preparation Blank" column.
- 3.4.5.2.8 For all blanks, enter the concentration (positive or negative) for each analyte, if the absolute value of the concentration is greater than or equal to the appropriate MDL. Enter the CRQL value for the analyte, if the absolute value of the concentration is less than the appropriate MDL.

For example, arsenic has a MDL of 3 ug/L for Preparation Method "NP1" [CRQL for arsenic is 10 ug/L (water)]. Therefore, a CCB instrument reading of -4.2485 ug/L will be reported as -4.249J; a CCB instrument reading of -2.4356 ug/L will be reported as 10.000U; a CCB instrument reading of 4.3586 ug/L will be reported as 4.359J; and a CCB instrument reading of 2.1584 ug/L will be reported as 10.000U.

3.4.5.2.9 Under "M", enter the method used, as explained in Exhibit B, Section 3.4.2.2.5.3.

- 3.4.5.2.10 If more than one wavelength/mass is used to analyze an analyte, submit additional Form(s) III-IN as appropriate.
- 3.4.5.2.11 The order of reporting ICBs and CCBs for each analyte shall follow the chronological order in which the blanks were run starting with the first Form III-IN and moving from left to right and continuing to additional Forms III-IN. When multiple wavelengths are used for the analysis of one analyte, all the results of one wavelength shall be reported before proceeding to the next wavelength.
- 3.4.6 ICP-AES and ICP-MS Interference Check Sample (ICS) [Forms IVA-IN and IVB-IN]
- 3.4.6.1 Purpose. These forms are used to report ICS results for each ICP-AES or ICP-MS instrument used in SDG analyses.
- 3.4.6.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions. The instructions for Forms IVA-IN and IVB-IN are identical except where specified.
- 3.4.6.2.1 For "ICP Instrument ID", enter an identifier that uniquely identifies a specific instrument within the Contractor laboratory. No two ICP instruments within a laboratory may have the same ICP Instrument ID.
- 3.4.6.2.2 Enter "ICS Source" (12 characters maximum) as explained in Exhibit B, Section 3.4.3.2.1. For USEPA solutions, include in the source name a number identifying it (e.g., EPA-LV87).
- 3.4.6.2.3 Under "True Sol. A", enter the true concentration (in ug/L, to two significant figures if the value is less than 10 and three significant figures if the value is greater than or equal to 10) of each analyte present in Solution A. Enter "0" for each analyte with no specified true value in Solution A.
- 3.4.6.2.4 Under "True Sol. AB", enter the true concentration (in ug/L, to two significant figures if the value is less than 10 and three significant figures if the value is greater than or equal to 10) of each analyte present in Solution AB. Enter "0" for each analyte with no specified true value in Solution AB.
- 3.4.6.2.5 Under "Initial Found Sol. A" on Form IVA-IN (ICP-AES), and "Found Sol. A" on Form IVB-IN (ICP-MS), enter the concentration (positive, negative, or zero, in ug/L, to two significant figures if the value is less than 10 and three significant figures if the value is greater than or equal to 10). Enter the concentration of each analyte and interferent for ICP-AES and of each analyte for ICP-MS in the initial analysis of Solution A as required in Exhibit D.
- 3.4.6.2.6 Under "Initial Found Sol. A %R" on Form IVA-IN (ICP-AES), and "Found Sol. A %R" on Form IVB-IN (ICP-MS), enter the value (to the nearest whole number) of the percent recovery computed for true Solution A greater than zero according to the following equation:

EQ. 5 Initial Found Sol. A Percent Recovery

$$%R = \frac{Initial Found Solution A}{True Found Solution A} \times 100$$

Leave the field blank if "True Solution A" equals zero.

- 3.4.6.2.7 Under "Initial Found Sol. AB" on Form IVA-IN (ICP-AES), and "Found Sol. AB" on Form IVB-IN (ICP-MS), enter the concentration (positive, negative, or zero, in ug/L, to two significant figures if the value is less than 10 and three significant figures if the value is greater than or equal to 10) of each analyte and interferent for ICP-AES and of each analyte for ICP-MS in the initial analysis of Solution AB as required in Exhibit D.
- 3.4.6.2.8 Under "Initial Found Sol. AB %R" on Form IVA-IN (ICP-AES), and "Found Sol. AB %R" on Form IVB-IN (ICP-MS), enter the value (to the nearest whole number) of the percent recovery computed for True Solution AB greater than zero according to the following equation:
 - EQ. 6 Initial Found Sol. AB Percent Recovery

$$R = \frac{\text{Initial Found Solution A}}{\text{True Solution A}} \times 100$$

Leave the field blank if "True Solution AB" equals zero.

- 3.4.6.2.9 Under "Final Found Sol. A", enter the concentration (positive, negative, or zero, in ug/L, to two significant figures if the value is less than 10 and three significant figures if the value is greater than or equal to 10) of each analyte and interferent for ICP-AES in the final analysis of Solution A as required in Exhibit D. ICP-MS analysis (Form IVB-IN) does not require a final analysis.
- 3.4.6.2.10 Under "Final Found Sol. A %R" enter the value (to the nearest whole number) of the percent recovery computed for true Solution A greater than zero according to the following equation:
 - EQ. 7 Final Found Sol. A Percent Recovery

$$R = \frac{\text{Final Found Solution A}}{\text{True Solution A}} \times 100$$

Leave the field blank if "True Solution A" equals zero.

3.4.6.2.11 Under "Final Found Sol. AB", enter the concentration (positive, negative, or zero, in ug/L, to two significant figures if the value is less than 10 and three significant figures if the value is greater than or equal to 10) of each analyte and interferent for ICP-AES in the final analysis of Solution AB as required in Exhibit D. ICP-MS analysis (Form IVB-IN) does not require a final analysis.

- 3.4.6.2.12 For all found values of Solutions A and AB, enter the concentration (positive, negative, or zero) of each analyte and interferent at each wavelength used for analysis by ICP.
- 3.4.6.2.13 Under "Final Found Sol. AB %R", enter the value (to the nearest whole number) of the percent recovery computed for true Solution AB greater than zero according to the following equation:
 - EQ. 8 Final Found Sol. AB Percent Recovery

$$R = \frac{\text{Final Found Solution AB}}{\text{True Solution AB}} \times 100$$

Leave the field empty if "True Solution AB" equals zero.

All percent recovery values reported shall be calculated using the exact true and found values reported on this form.

NOTE: For ICP-AES (Form IVA-IN), for every initial solution reported there must be a final solution reported. However, the opposite is <u>not</u> true. If an ICS was required to be analyzed in the middle of a run, it shall be reported in the "Final Found" section of this form.

- 3.4.6.2.14 If more ICS analyses were required, submit additional Form(s) IVA-IN and/or IVB-IN as appropriate.
- 3.4.6.2.15 The order of reporting ICSs for each analyte shall follow the chronological order in which the standards were run, starting with the first Form IVA-IN and/or IVB-IN and continuing to the following Forms IV-IN as appropriate. When multiple wavelengths/masses are used for one analyte, all the results of one wavelength/mass shall be reported before proceeding to the next wavelength/mass.
- 3.4.7 Matrix Spike Sample Recovery [Form VA-IN]
- 3.4.7.1 Purpose. This form is used to report results for the pre-digest spike.
- 3.4.7.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.
- 3.4.7.2.1 Indicate the appropriate matrix, level, and concentration units (ug/L for water and mg/kg dry weight for soil) as explained in Exhibit B, Sections 2.5.2.1.1 and 3.3.8.
- 3.4.7.2.2 For "% Solids for Sample", enter the percent solids (see Exhibit B, Section 3.4.2.2.2) for the original sample of EPA sample number reported on the form. Note that this number must equal the one reported on Form IA-IN for that sample.
- 3.4.7.2.3 In the "EPA Sample No." box, enter an EPA sample number (7 places maximum) of the sample from which the spike results on this form were obtained. The number shall be centered in the box.
- 3.4.7.2.4 Under "Control Limit R", enter "75-125" if the sample result is less than or equal to four times the spike added value. If

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the sample result is greater than four times the Spike Added (SA) value, leave this field empty.

- 3.4.7.2.5 Under "Spiked Sample Result (SSR)", enter the measured value (to four decimal places), in appropriate units, for each relevant analyte in the matrix spike sample. Enter the value of the result (if the concentration is greater than or equal to the MDL) corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, to the "C" qualifier column immediately following the "Spiked Sample Result (SSR)" column.
- 3.4.7.2.6 Under "Sample Result (SR)", enter the measured value (to four decimal places) for each required analyte in the sample (reported in "EPA Sample No." box) on which the matrix spike was performed. Enter the value of the result (if the concentration is greater than or equal to the MDL) corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions, if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, to the "C" qualifier column immediately following the "Sample Result (SR)" column.
- 3.4.7.2.7 Under "Spike Added (SA)", enter the value (to two decimal places) for the concentration of each analyte added to the sample. The same concentration units shall be used for "SSR", "SR", and "SA". If the "Spike Added" concentration is specified in the contract, the value added and reported shall be the specific concentration in appropriate units, corrected for spiked sample weight and percent solids (soils) or spiked sample volume (waters).
- 3.4.7.2.8 Under "%R", enter the value (to the nearest whole number) of the percent recovery for all spiked analytes computed according to the following equation:
 - EQ. 9 Spike Percent Recovery

$$R = \frac{SSR - SR}{SA} \times 100$$

Percent recovery shall be reported, whether it is negative, positive or zero.

The values for "SSR", "SR", and "SA" must be exactly those reported on this form. A value of zero shall be used in calculations for "SSR" or "SR" if the analyte value is less than the MDL.

- 3.4.7.2.9 Under "Q", enter "N" if the Spike Recovery (%R) is out of the control limits (75-125) and the Sample Result (SR) is less than or equal to four times the SA.
- 3.4.7.2.10 Under "M", enter the method used (as explained in Exhibit B, Section 3.4.2.2.5.3) or enter "NR" if the analyte is not required in the spike.

- 3.4.7.2.11 If different samples were used for spike sample analysis of different analytes, additional Form(s) VA-IN shall be submitted for each sample as appropriate.
- 3.4.8 Post-Digestion Spike Sample Recovery [Form VB-IN]
- 3.4.8.1 Purpose. This form is used to report results for the post-digest spike recovery which is based upon the addition of a known quantity of analyte to an aliquot of the <u>digested</u> sample.
- 3.4.8.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.
- 3.4.8.2.1 In the "EPA Sample No." box, enter an EPA sample number (seven characters maximum) of the sample from which the spike results on this form were obtained. The number shall be centered in the box.
- 3.4.8.2.2 The "Control Limit %R" and "Q" fields shall be left blank until limits are established by USEPA. At that time, the Contractor will be informed how to complete these fields.
- 3.4.8.2.3 Under "Spiked Sample Result (SSR)", enter the measured value (in ug/L, to two decimal places) for each analyte in the post-digest spike sample. Enter the value of the result (if the concentration is greater than or equal to the MDL); or enter the CRQL for the analyte if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, to the "C" qualifier column immediately following the "Spiked Sample Result (SSR)" column.
- 3.4.8.2.4 Under "Sample Result (SR)", enter the measured value (in ug/L, to two decimal places) for the concentration of each analyte in the sample (reported in "EPA Sample No." box) on which the spike was performed. Enter the value of the result (if the concentration is greater than or equal to the MDL); or enter the CRQL for the analyte if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, to the "C" qualifier column immediately following the "Sample Result (SR)" column.
- 3.4.8.2.5 Under "Spike Added (SA)", enter the value (in ug/L, to one decimal place) for each analyte added to the sample. If the SA concentration is specified in the contract, the value added and reported shall be that specific concentration in appropriate units.
- 3.4.8.2.6 Under "%R", enter the value (to the nearest whole number) of the percent recovery for all spiked analytes computed according to EQ. 9 in Exhibit B, Section 3.4.7.2.8. Percent recovery shall be reported, whether it is negative, positive, or zero. The values for "SSR", "SR", and "SA" must be exactly those reported on this form. A value of zero shall be substituted for "SSR" or "SR" if the analyte value is less than the MDL.
- 3.4.8.2.7 Under "M", enter the method used as explained in Exhibit B, Section 3.4.2.2.5.3, or enter "NR" if the spike was not required.

- 3.4.8.2.8 If different samples were used for spike sample analysis of different analytes, additional Form(s) VB-IN shall be submitted.
- 3.4.9 Duplicates [Form VI-IN]
- 3.4.9.1 Purpose. The duplicates form is used to report results of duplicate analyses. Duplicate analyses are required for percent solids values and all analyte results.
- 3.4.9.2 Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.
- 3.4.9.2.1 Indicate the appropriate matrix, level, and concentration units (ug/L for water and mg/kg dry weight for soil) as explained in Exhibit B, Sections 2.5.2.1.1 and 3.3.8.
- 3.4.9.2.2 For "% Solids for Sample", enter the percent solids (as explained in Exhibit B, Section 3.4.2.2.2) for the original sample of the EPA sample number reported on the form. Note that this number must equal the one reported on Form IA-IN for that sample.
- 3.4.9.2.3 For "% Solids for Duplicate", enter the percent solids (as explained in Exhibit B, Section 3.4.2.2.2) for the duplicate sample of the EPA sample number reported on the form.
- 3.4.9.2.4 In the "EPA Sample No." box, enter EPA sample number (seven characters maximum) of the sample from which the duplicate sample results on this form were obtained. The number shall be centered in the box.
- 3.4.9.2.5 Under "Control Limit", enter the CRQL (in appropriate units, ug/L for water or mg/kg dry weight basis corrected for the original sample weight and percent solids) for the analyte if either the sample or duplicate value was less than 5 times the CRQL. If the sample and duplicate values were greater than or equal to 5 times the CRQL, or if the sample and duplicate values were less than the CRQL, leave the field empty.
- 3.4.9.2.6 Under "Sample (S)", enter the original measured value (to four decimal places) for the concentration of each analyte in the sample (reported in "EPA Sample No." box) on which a duplicate analysis was performed. Concentration units are those specified on the form. Enter the value of the result (if the concentration is greater than or equal to the MDL) corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions, if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, to the "C" qualifier column immediately following the "Sample (S)" column.
- 3.4.9.2.7 Under "Duplicate (D)", enter the measured value (to four decimal places) for each analyte in the duplicate sample. Concentration units are those specified on the form. Enter the value of the result (if the concentration is greater than or equal to the MDL) corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions, if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in

Exhibit B, Section 3.4.2.2.5.1, to the "C" qualifier column immediately following the "Duplicate (D)" column.

- 3.4.9.2.8 For solid samples, the concentration of the original sample shall be computed using the weight and percent solids of the original sample. The concentration of the duplicate sample shall be computed using the weight of the duplicate sample, but the percent solids of the original sample.
- 3.4.9.2.9 Under "RPD", enter the absolute value (to the nearest whole number) of the Relative Percent Difference (RPD) for all analytes detected above the MDL in either the sample or the duplicate, computed according to the following equation:
 - EQ. 10 Duplicate Sample Relative Percent Difference

$$RPD = \frac{|S-D|}{(S+D)/2} \times 100$$

The values for "S" and "D" shall be exactly those reported on this form. A value of zero shall be substituted for "S" or "D" if the analyte concentration is less than the MDL in either one. If the analyte concentration is less than the MDL in both "S" and "D", leave the "RPD" field empty.

- 3.4.9.2.10 Under "Q", enter "*" if the duplicate analysis for the analyte is out of control. If both sample and duplicate values are greater than or equal to 5 times the CRQL, then the RPD must be less than or equal to 20% to be in control. If either the sample or duplicate value is less than 5 times the CRQL, then the absolute difference between the sample and duplicate values shall be less than the CRQL to be in control.
- 3.4.9.2.11 If both values are below the CRQL, then no control limit is applicable.
- 3.4.9.2.12 Under "M", enter method used as explained in Exhibit B, Section 3.4.2.2.5.3.
- 3.4.10 Laboratory Control Sample [Form VII-IN]
- 3.4.10.1 Purpose. This form is used to report results for the solid and aqueous LCSs.
- 3.4.10.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.
- 3.4.10.2.1 For the Solid LCS Source (12 characters maximum), enter the appropriate EPA sample number if EPA provided standard was used. Substitute an appropriate number provided by EPA for LCS solutions prepared in the future. If other sources were used, identify the source. For the aqueous LCS Source, enter the source name (12 characters maximum) as explained in Exhibit B, Section 3.4.3.2.1.
- 3.4.10.2.2 Under "Aqueous True", enter the value (in ug/L, to one decimal place) of the concentration of each analyte in the Aqueous LCS Standard Source.

- 3.4.10.2.3 Under "Aqueous Found", enter the measured concentration (in ug/L, to two decimal places) of each analyte found in the Aqueous LCS solution.
- 3.4.10.2.4 Under "Aqueous %R", enter the value of the percent recovery (to the nearest whole number) computed according to the following equation:
 - EQ. 11 Aqueous LCS Percent Recovery

$%R = \frac{\text{Solid LCS Found}}{\text{Solid LCS True}} \times 100$

- 3.4.10.2.5 Under "Solid True", enter the value (in mg/kg, to one decimal place) of the concentration of each analyte in the solid LCS Source.
- 3.4.10.2.6 Under "Solid Found", enter the measured value (in mg/kg, to one decimal place) of each analyte found in the solid LCS solution. Enter the value of the result (if the concentration is greater than or equal to the MDL) corrected for any dilutions; or enter the CRQL for the analyte, adjusted if necessary and corrected for any dilutions, if the concentration is less than the MDL.
- 3.4.10.2.7 Under "C", enter "J" or "U" or leave empty, to describe the found value of the solid LCS as explained in Exhibit B, Section 3.4.2.2.5.1.
- 3.4.10.2.8 Under "Limits", enter the lower limit (in mg/kg, to one decimal place) in the left column, and the upper limit (in mg/kg, to one decimal place) in the right column, for each analyte in the solid LCS solution.
- 3.4.10.2.9 Under "Solid %R", enter the value of the percent recovery (to the nearest whole number) computed according to the following equation:
 - EQ. 12 Solid LCS Percent Recovery

$%R = \frac{\text{Solid LCS Found}}{\text{Solid LCS True}} \times 100$

- 3.4.10.2.10 The values for true and found aqueous and solid LCSs used in EQs. 11 and 12 shall be exactly those reported on this form. If the analyte concentration is less than the MDL, a value of zero shall be substituted for the aqueous and solid LCS found.
- 3.4.10.2.11 Submit additional Form(s) VII-IN as appropriate if more than one aqueous LCS or solid LCS was required.
- 3.4.11 ICP-AES and ICP-MS Serial Dilutions [Form VIII-IN]
- 3.4.11.1 Purpose. This form is used to report results for ICP-AES and ICP-MS serial dilutions.
- 3.4.11.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.

- 3.4.11.2.1 In the "EPA Sample No." box, enter EPA sample number (7 places maximum) of the sample for which serial dilution analysis results on this form were obtained. The number shall be centered in the box.
- 3.4.11.2.2 Under "Initial Sample Result (I)", enter the instrument measured value (in ug/L to two decimal places) for each ICP analyte. This value shall not be corrected for any dilution. Enter the instrument measured value (if the concentration is greater than or equal to the MDL); or enter the CRQL if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, to the "C" qualifier column immediately following the "Initial Sample Result (I)" column.

NOTE: The initial sample concentration for an analyte does not have to equal the value for that analyte reported on Form IA-IN for that sample. It is the value of the analyte's instrument measured value (uncorrected for dilution) that is within the linear range of the instrument.

3.4.11.2.3 Under "Serial Dilution Result (S)", enter the instrument measured value corrected for a five-fold dilution (in ug/L to two decimal places) for each ICP analyte in the diluted sample. Enter the corrected instrument measured value (if the concentration is greater than or equal to the MDL); or enter the CRQL if the concentration is less than the MDL. Enter any appropriate concentration qualifier, as explained in Exhibit B, Section 3.4.2.2.5.1, to the "C" qualifier column immediately following the "Serial Dilution Result (S)" column.

NOTE: The "Serial Dilution Result (S)" is obtained by multiplying by five the instrument measured value (in ug/L) of the serially diluted sample. The "C" qualifier for the serial dilution shall be established based on the serial dilution result before correcting it for the five-fold dilution regardless of the value reported on Form VIII-IN.

For example, if the instrument readout value for the "Initial Sample Result (I)" for silver in a two-fold diluted sample MAX123 is 1164.36 ug/L, and the instrument readout value for the "Serial Dilution Result (S)" for silver in a ten-fold diluted sample MAX123 (MAX123L) is 241.67 ug/L, then the concentration reported for silver in the "Initial Sample Result (I)" column will be 1164.36 ug/L (not 2 times the instrument readout value which equals 2328.72 ug/L), and the concentration reported for silver in the "Serial Dilution Result (S)" column will be five times the instrument readout value which equals 1208.35 ug/L (not 10 times the instrument readout value which equals 2416.70 ug/L).

- 3.4.11.2.4 Under "% Difference", enter the absolute value (to the nearest whole number) of the percent difference in concentration of required analytes, between the original sample and the diluted sample (adjusted for dilution) according to the following formula:
 - EQ. 13 Serial Dilution Percent Difference

$$\text{%Difference} = \frac{|I - S|}{T} \times 100$$

The values for "I" and "S" used to calculate percent difference in EQ. 13 shall be exactly those reported on this form. A value of zero shall be substituted for "S" if the analyte concentration is less than the MDL. If the analyte concentration in (I) is less than the MDL concentration, leave the " $\$ Difference" field empty.

- 3.4.11.2.5 Under "Q", enter "E" if the percent difference is greater than 10% and the original sample concentration (reported on Form IA-IN) is greater than 50 times the MDL reported on Form IX-IN.
- 3.4.11.2.6 Under "M", enter the method of analysis for each analyte as explained in Exhibit B, Section 3.4.2.2.5.3.
- 3.4.12 Method Detection Limits (Annually) [Form IX-IN]
- 3.4.12.1 Purpose. This form documents the Method Detection Limits (MDLs) for each preparation method and instrument that the Contractor used to obtain data for the SDG. Only the methods, instruments, and wavelengths used to generate data for the SDG shall be included. The Contractor shall also report MDLs, obtained from direct analysis, for each instrument used to obtain data for the SDG. The MDLs obtained from direct analysis shall be used in the qualification of data associated with samples and instrument QC standards that are not taken through a preparation procedure. Although the MDLs are determined annually, a copy of the annual MDLs shall be included with each Sample Data Package on Forms IX-IN.
- 3.4.12.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.
- 3.4.12.2.1 Enter the Analysis Method qualifier as specified in Exhibit B, Section 3.4.2.2.5.3, in the "Instrument Type" field.
- 3.4.12.2.2 Enter the Instrument ID in the "Instrument ID" field (12 characters maximum). These instrument IDs are used to uniquely identify each instrument that the laboratory used to perform the analysis.
- 3.4.12.2.3 Enter the date (formatted MM/DD/YYYY) on which the MDL analysis was performed in the "Date" field.
- 3.4.12.2.4 For "Preparation Method", enter the method of preparation (three characters maximum) for which the MDLs listed on Form IX-IN were established. Use appropriate sample preparation codes as specified below:

- HW1: Hotplate/Block digestion for ICP-AES analysis of water samples.
- HW2: Hotplate/Block digestion for ICP-MS analysis of water samples.
- HW3: Hotplate/Block digestion for ICP-MS analysis of water samples.
- MW1: Microwave digestion for ICP-AES analysis of water samples.
- MW2: Microwave digestion for ICP-AES analysis of water samples.
- HS1: Hotplate/Block digestion for ICP-AES analysis of soil samples.
- HS2: Hotplate/Block digestion for ICP-AES analysis of soil samples.
- MS1: Microwave digestion for ICP-AES analysis of soil samples.
- CW1: Preparation for the Manual Cold Vapor AA analysis of water samples.
- CS1: Preparation for the Manual Cold Vapor AA analysis of soil samples.
- CW2: Preparation for the Automated Cold Vapor AA analysis of water samples.
- DW1: Distillation for the manual and semi-automated spectrophotometric analysis of water samples.
- DW2: Midi-distillation for the semi-automated spectrophotometric analysis of water samples.
- DS1: Distillation for the manual and semi-automated spectrophotometric analysis of soil samples.
- DS2: Midi-distillation for the semi-automated spectrophotometric analysis of soil samples.
- NP1: No preparation.
- 3.4.12.2.5 Enter the concentration units (UG/L for water or MG/KG wet weight for soil) for the results reported on Form IX-IN in the "Concentration Units" field. Enter "UG/L" for MDL results obtained from direct analysis (Preparation Method "NP1").
- 3.4.12.2.6 Under "Wavelength/Mass", enter the wavelength in nanometers (nm) to two decimal places or the mass in atomic mass units (amu) to two decimal places for each analyte for which an MDL has been established and is listed in the MDL column. If more than one wavelength or mass is used for an analyte, use additional Form(s) IX-IN as appropriate to report the MDL.
- 3.4.12.2.7 Contract Required Quantitation Limits (in ug/L for water and mg/kg for soil) as established in Exhibit C, shall be reported in the column headed "CRQL". The CRQL shall be reported in ug/L on Form(s) IX-IN associated with Preparation Method "NP1".
- 3.4.12.2.8 Under "MDL", enter the MDL (in ug/L for water and direct analysis, or mg/kg for soil, to two significant figures for values less than 10, and three significant figures for values greater than or equal to 10) as determined by the Contractor for each analyte analyzed by the instrument for which the ID is listed on this form. When calculating MDL values, always round up to the appropriate significant figure (e.g., 14.81 rounds to 14.9 and 146.6 rounds to 147). This deviation from the rounding rule is necessary to prevent the reporting of detected values for results that fall in the noise region of the calibration curve.

NOTE: Zeros used to set the decimal point in a number less than one are not significant but all trailing zeros are significant.

For example, a calculated MDL value of 0.074~ug/L will be reported as 0.074~and a calculated MDL value of 0.1~or~0.08 will be reported as 0.10~and~0.080, respectively.

- 3.4.12.2.9 Use additional Form(s) IX-IN if more preparation methods, instruments and wavelengths or masses are used. Note that the date on this form shall not exceed the analysis dates in the Sample Data Package or precede them by more than twelve months.
- 3.4.12.2.10 Use the "Comments" section to indicate alternative wavelengths and the conditions under which they are used.
- 3.4.13 ICP-AES Interelement Correction Factors (Quarterly) [Form XA-IN]
- 3.4.13.1 Purpose. This form documents for each ICP-AES instrument the interelement correction factors applied by the Contractor to obtain data for the SDG. Although the correction factors are determined quarterly, a copy of the results of the quarterly interelement correction factors shall be included with each Sample Data Package on Form XA-IN and Form XB-IN as appropriate.
- 3.4.13.2 Instructions. Complete the header information according to instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.
- 3.4.13.2.1 Enter the ICP-AES Instrument ID (12 characters maximum), which is a unique number designated by the Contractor to identify each ICP-AES instrument used to produce data in the Sample Data Package. If more than one ICP-AES instrument is used, submit additional Form(s) XA-IN as appropriate.
- 3.4.13.2.2 Report the date (formatted as MM/DD/YYYY) on which these correction factors were determined for use. This date shall not exceed the ICP-AES analysis dates in the Sample Data Package or precede them by more than three calendar months.
- 3.4.13.2.3 Under "Wavelength", list the wavelength in nm (to two decimal places) used for each ICP-AES analyte. If more than one wavelength is used, submit additional Form(s) XA-IN or Form(s) XB-IN as appropriate.
- 3.4.13.2.4 Under "Al", "Ca", "Fe", and "Mg", enter the correction factor (negative, positive or zero, to seven decimal places, 10 characters maximum) for each ICP-AES analyte. Correction factors for one other analyte shall be reported using the empty column and listing the analyte's chemical symbol in the blank two-space header field provided for that column.
- 3.4.13.2.5 If corrections are not applied for an analyte, a zero shall be entered for that analyte to indicate that the corrections were determined to be zero. Correction factors for more than one additional analyte shall be reported using Form XB-IN.

NOTE: Correction factors for Al, Ca, Fe, and Mg are all required and are to be listed first (as they appear on Form XA-IN).

- 3.4.14 ICP-AES Interelement Correction Factors (Quarterly) [Form XB-IN]
- 3.4.14.1 Purpose. This form is used if correction factors for analytes other than Al, Ca, Fe, Mg, and one more analyte of the Contractor's choice were applied to the analytes analyzed by ICP-AES.
- 3.4.14.2 Instructions. Complete this form following the instructions for Form XA-IN (see Exhibit B, Section 3.4.13) by listing the chemical symbol for additional analytes in the heading of the empty columns in the two-space fields provided.
- 3.4.14.2.1 Columns of correction factors for additional analytes shall be entered left to right starting on Form XA-IN and proceeding to Form XB-IN, according to the alphabetical order of their chemical symbols.
- 3.4.15 ICP-AES and ICP-MS Linear Ranges (Quarterly) [Form XI-IN]
- 3.4.15.1 Purpose. This form documents the quarterly linear range analysis for each ICP instrument that the Contractor used to obtain data for the SDG.
- 3.4.15.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.
- 3.4.15.2.1 Enter the ICP Instrument ID (12 characters maximum), which is a unique number designated by the Contractor to identify each ICP instrument used to produce data for the SDG. If more than one ICP instrument is used, submit additional Form(s) XI-IN as appropriate.
- 3.4.15.2.2 Report the date (formatted as MM/DD/YYYY) on which these linear ranges were analyzed. This date shall not exceed the dates of analysis by ICP in the Sample Data Package and shall not precede the analysis dates by more than three calendar months.
- 3.4.15.2.3 Under "Integ. Time (Sec.)", enter the integration time (in seconds to two decimal places) used for each measurement taken from the ICP instrument.
- 3.4.15.2.4 Under "Concentration", enter the concentration (in ug/L) that is the upper limit of the ICP instrument linear range as determined in Exhibit D. Any measurement above it is out of the linear range, and thus, is an estimated value and shall be diluted into the linear range.
- 3.4.15.2.5 Under "M", enter the method of analysis for each analyte as explained in Exhibit B, Section 3.4.2.2.5.3.
- 3.4.15.2.6 If more instruments or analyte wavelengths/masses are used, submit additional Form(s) XI-IN as appropriate.
- 3.4.16 Preparation Log [Form XII-IN]
- 3.4.16.1 Purpose. This form is used to report the preparation run log.
- 3.4.16.1.1 All field samples and all Quality Control (QC) preparations (including duplicates, matrix spikes, LCSs, PBs, and repreparations) associated with the SDG shall be reported on Form XII-IN. In addition, for mercury analyses, all prepared

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Form Instructions
Form XIII-IN

calibration standards and QC standards (e.g., ICV, CCV, ICB, CCB, CRI) shall also be reported on Form XII-IN. For cyanide analyses, the distilled ICV and the mid-range standard shall also be reported on Form XII-IN.

- 3.4.16.1.2 Submit one Form XII-IN per batch, per method, if no more than thirty-two preparations, including QC preparations, were performed. If more than 32 preparations per batch, per method, were performed, then submit additional copies of Form XII-IN as appropriate. Submit a separate Form XII-IN for each batch.
- 3.4.16.1.3 The order in which the Preparation Logs are submitted is very important. Form XII-IN shall be organized by method, by batch. Later batches within a method shall follow earlier ones. Each batch shall start on a separate Form XII-IN.
- 3.4.16.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.
- 3.4.16.2.1 For "Preparation Method", enter the method of preparation (three characters maximum) for which the preparations listed on Form XII-IN were made, as specified in Exhibit B, Section 3.4.12.2.4.
- 3.4.16.2.2 Under "EPA Sample No.", enter EPA sample number of each sample in the SDG, and of all other preparations such as duplicates, matrix spikes, LCSs, PBs, and re-preparations (all formatted according to Exhibit B, Table 2). All EPA sample numbers shall be listed in ascending alphanumeric order, continuing to the next Form XII-IN if applicable.
- 3.4.16.2.3 Under "Preparation Date", enter the date (formatted MM/DD/YYYY) on which each sample was prepared for analysis by the method indicated in the header section of the form.

NOTE: The date never changes on a single Form XII-IN because the form shall be submitted per batch.

- 3.4.16.2.4 Under "Weight", enter the wet weight (in grams, to two decimal places) of each soil sample prepared for analysis by the method indicated in the header section of the form. If the sample matrix is water, then leave the field empty.
- 3.4.16.2.5 Under "Volume", enter the final volume (in mL, to the nearest whole number) of the preparation for each sample prepared for analysis by the method indicated in the header section of the form. This field shall have a value for each sample listed.
- 3.4.17 Analysis Run Log [Form XIII-IN]
- 3.4.17.1 Purpose. This form is used to report the sample analysis run log.
- 3.4.17.1.1 A run is defined as the totality of analyses performed by an instrument throughout the sequence initiated by, and including, the first SOW-required calibration standard or tune standard, and terminated by, and including, the CCV and CCB following the last SOW-required analytical sample.
- 3.4.17.1.2 All field samples and all QC analyses (including tunes, calibration standards, ICVs, CCVs, ICBs, CCBs, CRIs, ICSs, LRSs, LCSs, PBs, duplicates, serial dilutions, matrix spikes,

and post-digestion/distillation spikes) associated with the SDG shall be reported on Form XIII-IN. The run shall be continuous and inclusive of all analyses performed on the particular instrument during the run.

- 3.4.17.1.3 Submit one Form XIII-IN per run if no more than thirty-two (32) analyses, including instrument calibration, were analyzed in the run. If more than thirty-two analyses were performed in the run, submit additional Form(s) XIII-IN as appropriate.
- 3.4.17.1.4 The order in which the Analysis Run Logs are submitted is very important. Form XIII-IN shall be organized by method, and by run. Later runs within a method shall follow earlier ones. Each analytical run shall start on a separate Form XIII-IN. Therefore, instrument calibration or tune shall be the first entry on the form for each new run. In addition, the run is considered to have ended if it is interrupted for any reason, including termination for failing QC parameters.
- 3.4.17.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.
- 3.4.17.2.1 For "Instrument ID", enter the Instrument ID (12 characters maximum) which shall be an identifier designated by the Contractor to uniquely identify each instrument used to produce data which are required to be reported in the SDG deliverable. If more than one instrument is used, submit additional Form(s) XIII-IN as appropriate. The Instrument ID shall exactly match that reported on Forms IVA, IVB, IX, XA, XB, XI, XIV, and XV.
- 3.4.17.2.2 For "Analysis Method", enter the method code (two characters maximum) according to the specifications in Exhibit B, Section 3.4.2.2.5.3.
- 3.4.17.2.3 For "Start Date", enter the date (formatted MM/DD/YYYY) on which the analysis run was started.
- 3.4.17.2.4 For "End Date", enter the date (formatted MM/DD/YYYY) on which the analysis run was ended.
- 3.4.17.2.5 Under "EPA Sample No.", enter EPA sample number of each analysis, including all QC operations applicable to the SDG (formatted according to Exhibit B, Table 2). All EPA sample numbers shall be listed in increasing chronological (date and time) order of analysis, continuing to the next Form XIII-IN for the instrument run, if applicable. The analysis date and time of other analyses not associated with the SDG, but analyzed by the instrument in the reported analytical run, shall be reported. Those analyses shall be identified with EPA sample number of "ZZZZZZ".
- 3.4.17.2.6 Under "D/F", enter the dilution factor (to two significant figures) by which the final digestate or distillate needed to be diluted for each analysis to be performed. The dilution factor does not include the dilution inherent in the preparation as specified by the preparation procedures in Exhibit D.
- 3.4.17.2.7 The dilution factor is required for all entries on Form XIII- $_{
 m IN}$.

NOTE: For a particular sample a dilution factor of "1.0" shall be entered if the digestate or distillate was analyzed without adding any further volume of dilutant or any other solutions to the "Volume" or an aliquot of the "Volume" listed on Form XII-IN for that sample.

- 3.4.17.2.8 For USEPA supplied solutions such as ICVs, ICSs, and LCSs, a dilution factor shall be entered if the supplied solution had to be diluted to a dilution different from that specified by the instructions provided with the solution. The dilution factor reported in such a case shall be that which would make the reported true values on the appropriate form for the solution equal those that were supplied with the solution by USEPA. For instance, ICV-2(0887) has a true value of 104.0 ug/L at a 20-fold dilution. If the solution is prepared at a 40-fold dilution, a dilution factor of "2.0" shall be entered on Form XIII-IN and the uncorrected instrument reading is compared to a true value of 52 ug/L. In this example, Form IIA-IN will have a true value of 104.0 regardless of the dilution used. The found value for the ICV shall be corrected for the dilution listed on Form XIII-IN using the following formula:
 - EQ. 14 ICV/CCV Correction for Dilution

Found value on Form II = Instrument readout $(uq/L) \times D/F$

- 3.4.17.2.9 Under "Time", enter the time (in military format HHMM) at which each analysis was performed.
- 3.4.17.2.10 Under "Analytes", enter "X" in the column of the designated analyte to indicate that the analyte value was used from the reported analysis to report data in the SDG. Leave the column empty for each analyte if the analysis was not used to report the particular analyte.
- 3.4.17.2.11 Entering "X" appropriately is very important. The "X" is used to link the samples with their related QC. It also links the dilution factor with the appropriate result reported on Inorganic Forms I-VIII. For each analyte result reported on any of the Forms I-VIII, there shall be one, and only one, properly identified entry on Form XIII-IN for which an "X" is entered in the column for that analyte.
- 3.4.17.2.12 If, on Form XIII-IN, an "X" is entered in the column for an analyte for a field sample associated with a dilution factor greater than 1.0, flag the data for that analyte with a "D" on the appropriate Form IA-IN or Form IB-IN.
- 3.4.18 ICP-MS Tune [Form XIV-IN]
- 3.4.18.1 Purpose. This form is used to report the tuning results for each ICP-MS instrument used in SDG analyses.
- 3.4.18.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.
- 3.4.18.2.1 For "ICP-MS Instrument ID", enter an identifier that uniquely identifies a specific instrument within the Contractor

laboratory. No two ICP-MS instruments within a laboratory may have the same ICP-MS Instrument ID.

- 3.4.18.2.2 Report the date (formatted as MM/DD/YYYY) on which the ICP-MS tune was performed. This date shall not exceed the dates of analysis by ICP-MS in the Sample Data Package.
- 3.4.18.2.3 For "Avg. Measured Mass (amu)", enter the average mass calculated from the five or more tune analyses (in atomic mass units, to two decimals places) measured for each isotope.
- 3.4.18.2.4 For "Avg. Peak Width at Peak Height (amu)" enter the average peak width calculated from the analysis (in atomic mass units, to two decimal places) at the percent of peak height recommended by the instrument manufacturer for each isotope.
- 3.4.18.2.5 For "%RSD", enter the percent Relative Standard Deviation of the absolute signals (intensities) for each isotope calculated from the five or more tune analyses.
- 3.4.19 ICP-MS Internal Standards Relative Intensity Summary [Form XV-IN]
- 3.4.19.1 Purpose. This form is used to report the relative internal standard intensity levels during a run for ICP-MS. The relative intensity of each of the internal standards in all analyses performed by ICP-MS must be reported on the form. If more than one ICP-MS instrument or run is used, submit additional Form(s) XV-IN as appropriate. All runs for the lowest alphanumeric instrument must be reported in ascending order before proceeding to the runs for the next highest instrument.
- 3.4.19.2 Instructions. Complete the header information according to the instructions in Exhibit B, Section 3.3. Complete the remainder of the form using the following instructions.
- 3.4.19.2.1 For "ICP-MS Instrument ID", enter an identifier that uniquely identifies a specific instrument within the Contractor laboratory. No two ICP-MS instruments within a laboratory may have the same ICP-MS Instrument ID.
- 3.4.19.2.2 For "Start Date", enter the date (formatted MM/DD/YYYY) on which the analysis run was started.
- 3.4.19.2.3 For "End Date", enter the date (formatted MM/DD/YYYY) on which the analysis run was ended.
- 3.4.19.2.4 Under "EPA Sample No.", enter EPA sample number of each analysis, including all QC operations applicable to the SDG. All EPA sample numbers must be listed in increasing chronological (date and time) order of analysis, continuing to the next Form XV for the instrument run, if applicable. The order must agree with the order reported on Form XIII-IN for that run. The analysis date and time of other analyses not associated with the SDG, but analyzed by the instrument in the reported analytical run, must be reported. Those analyses must be identified with EPA sample number of "ZZZZZZZ." Samples identified as "ZZZZZZZ" need not have intensities reported for internal standards.
- 3.4.19.2.5 Under "Time", enter the time (in military format HHMM) at which each analysis was performed.

Exhibit B -- Section 3
Form Instructions
Form DC-1

- 3.4.19.2.6 Under "Internal Standards %RI for:", enter the chemical symbol and elemental expression number of the internal standard in the "Element" header field provided to indicate the internal standard and elemental expression for which the Relative Intensity (RI) of the internal standards will be calculated in that column.
- 3.4.19.2.6.1 In the "Element" column, enter the internal standard relative intensity (to the nearest whole number) of the internal standard for each sample analysis listed on the form (excluding "ZZZZZZZ"). The internal standard relative intensity (%RI) is calculated using the following formula:
 - EQ. 15 Internal Standard Percent Relative Intensity

$$%RI = \frac{I_n}{I_0} \times 100$$

WHERE, "I $_{\rm o}$ " is the intensity of the internal standard in the blank calibration standard and "I $_{\rm n}$ " is the intensity of the internal standard in the EPA sample number in the same units.

- 3.4.19.2.7 Under the "Q" column to the right of each "Element" column, enter an "R" if the %RI for a field sample, PE, duplicate, or spike is less than 60 or greater than 125; otherwise leave the field blank.
- 3.4.19.2.8 Columns of internal standard RI must be entered left to right starting with the internal standards of the lower mass on the first Form XV-IN and proceeding to the following Form XV-IN as appropriate. All Forms XV-IN for the lowest numeric instrument must be reported in ascending order by the run number before proceeding to the next Form XV.
- 3.4.19.3 All field samples and all QC samples (including calibration standards, ICVs, CCVs, ICBs, CCBs, CRIs, ICSs, LCS, PB, serial dilutions, duplicates, PE samples, and spikes) associated with the SDG must be reported on Form XV-IN. The run must be continuous and inclusive of all analyses performed on the particular instrument during the run.
- 3.4.19.4 Submit one Form XV-IN per run if no more than 32 analyses, including instrument calibration, were analyzed in the run. If more than 32 analyses were performed in the run, submit additional Form(s) XV-IN as appropriate. Each new run must be started on the first line of Form XV-IN.
- 3.5 Sample Log-In Sheet [Form DC-1]
- 3.5.1 Purpose. This form is used to document the receipt and inspection of samples and containers. At least one original Form DC-1 is required for each sample shipping container (e.g., cooler). If the samples in a single sample shipping container must be assigned to more than one SDG, the original Form DC-1 shall be placed with the deliverables for the SDG of the lowest alpha-numeric number and a copy of Form DC-1 shall be placed with the deliverables for the other SDG(s). The copies should be identified as "copy(ies)", and the location of the original should be noted on the copies.

- 3.5.2 Instructions
- 3.5.2.1 Sign and date the airbill. (If an airbill is not received, include a hardcopy receipt requested from the shipping company or a printout of the shipping company's electronic tracking information).
- 3.5.2.2 Examine the shipping container and record the presence/absence of custody seals and their condition (i.e., intact, broken) in Item 1.
- 3.5.2.3 Record the custody seal numbers in Item 2.
- 3.5.2.4 Open the container, remove the enclosed sample documentation, and record the presence/absence of USEPA forms (i.e., Traffic Reports/Chain of Custody Records, packing lists) and airbills or airbill stickers in Items 3 and 4. Specify if there is an airbill present or an airbill sticker in Item 4. Record the airbill or sticker number in Item 5.
- 3.5.2.5 Remove the samples from the shipping container(s), examine the samples and the sample tags (if present), and record the condition of the sample bottles (i.e., intact, broken, leaking) and presence or absence of sample tags in Items 6 and 7.
- 3.5.2.6 Record the presence or absence of a cooler temperature indicator bottle in Item 8.
- 3.5.2.7 Record the cooler temperature in Item 9.
- 3.5.2.8 Review the sample shipping documents and compare the information recorded on all the documents and samples and mark the appropriate answer in Item 10.
- 3.5.2.9 The log-in date should be recorded at the top of Form DC-1; record the date and time of cooler receipt at the laboratory in Items 11 and 12.
- 3.5.2.10 If there are no problems observed during receipt, sign and date (include the time) Form DC-1 and Traffic Report/Chain of Custody Record, and write the sample numbers in the "EPA Sample No." column.
- 3.5.2.11 Record the pH for all aqueous samples received.
- 3.5.2.12 Record the appropriate sample tags and assigned laboratory numbers, if applicable.
- 3.5.2.13 Any comments should be made in the "Remarks" column.
- 3.5.2.14 Record the fraction designation (if appropriate) and the specific area designation (e.g., refrigerator number) in the "Sample Transfer" block located in the bottom left corner of Form DC-1. Sign and date the sample transfer block.
- 3.5.2.15 For Items 1, 3, 4, 6, 7, 8 and 10, circle the appropriate response. Responses can be underlined if this form is completed by automated equipment. Unused columns and spaces shall be crossed out, initialed, and dated.
- 3.5.2.16 If there are problems observed during receipt (including samples that have not been preserved to the proper pH) or an answer marked

with an asterisk (e.g., "absent*") was circled, contact SMO and document the contact as well as resolution of the problem on a CLP Communication Log. Following resolution, sign and date the forms as specified in the preceding paragraph and note, where appropriate, the resolution of the problem.

- 3.6 Full Inorganics Complete SDG File (CSF) Inventory Sheet [Form DC-2]
- 3.6.1 Purpose. The CSF Inventory Sheet is used to record both the inventory of Complete SDG File (CSF) documents and the number of documents in the original Sample Data Package which is sent to the USEPA Region.
- 3.6.2 Instructions
- 3.6.2.1 Organize all EPA-CSF documents as described in Exhibit B, Sections 2 and 3. Assemble the documents in Exhibit B, Section 2 in the order specified on Form DC-2, and stamp each page with the consecutive number. Inventory the CSF by reviewing the document numbers and recording page number ranges in the columns provided on Form DC-2. The Contractor shall verify and record in the "Comments" section on Form DC-2 all intentional gaps in the page numbering sequence (for example, "page numbers not used, XXXX-XXXX, XXXX-XXXX"). If there are no documents for a specific document type, enter an "NA" in the empty space.
- 3.6.2.2 Certain laboratory-specific documents related to the CSF may not fit into a clearly defined category. The laboratory should review Form DC-2 to determine if it is most appropriate to place them under Categories 33, 34, 35, or 36. Category 36 should be used if there is no appropriate previous category. These types of documents should be described or listed in the blanks under each appropriate category.

B - 48

- 3.6.2.3 If it is necessary to insert new or inadvertently omitted documents, the Contractor shall follow these steps:
 - Number all documents to be inserted with the next sequential numbers and file the inserts in their logical positions within the CSF (e.g., file document 1000 between documents 6 and 7).
 - Identify where the inserts are filed in the CSF by recording the document numbers and their locations under the "Other Records" section of Form DC-2 (e.g., document 1000 is filed between 6 and 7).

4.0 DATA REPORTING FORMS

The data reporting forms are shown on the following pages.

EXHIBIT B

INORGANIC FORMS

COVER PAGE

Lab Name:		Con	tract:		
Lab Code:	Case No.:	NRAS No.: _	:	SDG No.: _	
SOW No.:					
	EPA Sample No.		Lab	Sample ID	
				ICP-AES	ICP-MS
Were ICP-AES corrections a	and ICP-MS intere	lement	(Yes/No)		
Were ICP-AES applied?	and ICP-MS backgr	ound corrections	(Yes/No)		
	re raw data gener n of background c		(Yes/No)		
Comments:					
conditions of than the conditation hardcopy data (or via an alter by USEPA) has been seen as the conditions of the condi	the contract, bot tions detailed ab package and in th ernate means of e peen authorized b	e is in compliance h technically and ove. Release of tecomputer-readablectronic transmisy the Laboratory Mallowing signature.	for complement data contact data submission, if applications or see the contact data and the	teness, fontained in mitted on oproved in	this diskette advance
Signature: Date:		Name: Title:			
		<u>-</u>			

		INORGA	1A-IN NIC ANALYSIS DA	TA SHEE'		EPA SAMPLE NO
Lab Name:	:		Contract:			
Lab Code:		Case No.: _	NRAS No	.:	SDG N	10.:
Matrix: ((soil/water)	Lab	Sample	ID:	
Level: (1	_ow/med)		Date	e Receiv	red:	
% Solids:		-				
Concentra F	ation Units	(ug/L or mo	g/kg dry weight):		
	CAS No.	Analyte	Concentration	С	Q	M
	7429-90-5					
L	7440-36-0	_				
L	7440-38-2					
L	7440-39-3	Barium				
<u> </u>		Beryllium				
	7440-43-9					
-	7440-70-2					
-	7440-47-3					
-	7440-48-4					
-	7440-50-8					
-	7439-89-6	Iron				
-	7439-92-1	Lead				
⊦		Magnesium				
	7439-96-5 7439-97-6					
F	7440-02-0	Nickel				
⊦	7440-09-7					
⊢	7782-49-2					
⊢	7440-22-4					
F	7440-23-5					
F	7440-28-0	Thallium				
F	7440-62-2	Vanadium				
F	7440-66-6	Zinc				
F	57-12-5	Cyanide				
F	0, 12 0	o y anni a d				
-						
Color Bef	fore:	Cla:	rity Before:		Texture:	
Color Aft	ter:	Cla	rity After:		Artifact	:s:

FORM IA-IN

Comments:

			USEPA - CLP					
		INORG	1B-IN ANIC ANALYSIS DA	TA SI	HEET	E	PA SA	MPLE NO.
Lab Name	:		Contract:					
Lab Code	:	Case No.: _	NRAS No	·:		SDG No	·: <u> </u>	
Matrix:	(soil/water	c)	Lab	Samp	le ID: _			
Level: ((low/med)		Date	e Rec	eived: _			
% Solids	s:	<u> </u>						
Concentr	ation Units	s (ug/L or m	ng/kg dry weight):				
	CAS No.	Analyte	Concentration	С	Q		М	

Color Before:	Clarity Before:	Texture:
Color After:	Clarity After:	Artifacts:
Comments:		

2A-IN INITIAL AND CONTINUING CALIBRATION VERIFICATION

Lab Name:		Contract:	
Lab Code:	Case No.:	NRAS No.:	SDG No.:
Initial Calibration	Verification Sour	ce:	
Continuing Calibrat	ion Verification So	ource:	
Concentration Units	: ug/L		

		al Calib rificati		Continuing Calibration Verification					M
Analyte	True	Found	%R(1)	True	Found	%R(1)	Found	%R(1)	
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									
_									

(1) Control Limits: Mercury 80-120; Other Metals 90-110; Cyanide 85-115

2B-IN CRQL CHECK STANDARD

Lab Name: _		Contrac	ct:	
Lab Code: _	Case No.:	NRAS No.:	SDG No.:	
CRQL Check	Standard Source:			
Concentrati	lon Units: ug/L			

	CRQL Check Standard									
	Init	ial		Final						
Analyte	True	Found*	%R(1)	Found*	%R(1)					
Aluminum										
Antimony										
Arsenic										
Barium										
Beryllium										
Cadmium										
Calcium										
Chromium										
Cobalt										
Copper										
Iron										
Lead										
Magnesium										
Manganese										
Mercury										
Nickel										
Potassium										
Selenium										
Silver										
Sodium										
Thallium										
Vanadium										
Zinc										
Cyanide										

⁽¹⁾ Control Limits: 70-130 with the following exceptions: ICP-AES - Antimony, Lead, and Thallium: 50-150. ICP-MS - Cobalt, Manganese, and Zinc: 50-150.

^{*} If applicable, enter the concentration qualifier "J" or "U" after the concentration in these columns (e.g., 0.20U for Mercury).

3-IN BLANKS

Lab Name:		Contract:	
Lab Code: _	Case No.:	NRAS No.:	SDG No.:
Preparation	Blank Matrix (soil/water)	:	
Drenaration	Blank Concentration Units	(ua/I or ma/ka)·	

	Initial Calibrati Blank (ug,	on		Continuing Calibration Blank (ug/L)					Preparation Blank		
Analyte		С	1	С	2	С	3	С		С	М
Aluminum											
Antimony											
Arsenic											
Barium											
Beryllium											
Cadmium											
Calcium											
Chromium											
Cobalt											
Copper											
Iron											
Lead											
Magnesium											
Manganese											
Mercury											
Nickel											
Potassium											
Selenium											
Silver											
Sodium											
Thallium											
Vanadium											
Zinc											
Cyanide			_		_		_				

4A-IN ICP-AES INTERFERENCE CHECK SAMPLE

Lab Name:	Contract:
Lab Code: Case No.: NRAS N	Jo.: SDG No.:
ICP-AES Instrument ID:	ICS Source:
Concentration Units: ug/L	

	Tr	ue		Initial	Found		Final Found			
Analyte	Sol. A	Sol. AB	Sol. A	%R	Sol. AB	%R	Sol. A	%R	Sol. AB	%R
Aluminum										
Antimony										
Arsenic										
Barium										
Beryllium										
Cadmium										
Calcium										
Chromium										
Cobalt										
Copper										
Iron										
Lead										
Magnesium										
Manganese										
Nickel										
Potassium										
Selenium										
Silver										
Sodium										
Thallium										
Vanadium										
Zinc										

4B-IN ICP-MS INTERFERENCE CHECK SAMPLE

Lab Name:	Contract:				
Lab Code:	_ Case No.:	NRAS No.:	SDG No.:		
ICP-MS Instrument	ID:	ICS Source:			

Concentration Units: ug/L

	True		Found			
Analyte	Sol. A	Sol. AB	Sol. A	%R	Sol. AB	%R
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
Carbon						
Chloride						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Molybdenum						
Nickel						
Phosphorus						
Potassium						
Selenium						
Silver						
Sodium						
Sulfur						
Thallium						
Titanium						
Vanadium		_				
Zinc						

5A-IN	EPA SAMPLE NO.
MATRIX SPIKE SAMPLE I	RECOVERY
Lab Name: Contract:	
Lab Code: Case No.: NRAS No	.: SDG No.:
Matrix: (soil/water)	Level: (low/med)
% Solids for Sample:	
Concentration Units (ug/L or mg/kg dry weight)):

Analyte	Control Limit %R	Spiked Sample Result (SSR)		Sample Result (SR	R.) C	Spike Added (SA)	%R	Q	М
	010		_	_			010	Ž	
Aluminum									-
Antimony									ļ
Arsenic									-
Barium									ļ
Beryllium									ļ
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

Comm	ents:				

5B-TN

		5B-IN	EPA SAMPLE NO.	
	POST-DIGESTIC	ON SPIKE SAMPLE RECC	OVERY	
Lab Name:		Contract:		
Lab Code:	Case No.:	NRAS No.:	SDG No.:	
Matrix: (soil/wa	ater)		Level: (low/med)	

Concentration Units: ug/L

Analyte	Control Limit %R	Spiked Sample Result (SSR)	Sample Result (S	R) C	Spike Added (SA)	%R	Q	M
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Vanadium								
Zinc								
Cyanide								

Comme	nts:			
_				
_				

FORM VB-IN

	EPA SAMPLE NO.	
	DUPLICATES	
Lab Name:	Contract:	
Lab Code: Case No.:	NRAS No.:	SDG No.:
Matrix: (soil/water)		Level: (low/med)
% Solids for Sample:	% Sol	ids for Duplicate:
Concentration Units (ug/I or mg/	/ka dry woight).	

Concentration Units (ug/L or mg/kg dry weight): ___

Analyte	Control Limit	Sample (S)	С	Duplicate (D)	С	RPD	Q	М
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Vanadium								
Zinc								
Cyanide								

7-IN LABORATORY CONTROL SAMPLE

Lab Name:		:		
Lab Code:	Case No.:	NRAS No.:	SDG No.:	
Solid LCS So	urce:			
Aqueous LCS	Source:			

	Aque	eous (ug/L)		Solid (mg/kg)					
Analyte	True	Found	%R	True	Found	С	Lim	its	%R
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									
_									

8-IN

		8-IN	EPA SAMPLE NO.
	ICP-AES and	ICP-MS SERIAL DILU	JTIONS
Lab Name: _		_ Contract:	
Lab Code: _	Case No.:	NRAS No.:	SDG No.:
Matrix: (so	oil/water)		Level: (low/med)

Concentration Units: ug/L

Analyte	Initial Sample Result (I) C	Serial Dilution Result (S) C	% Difference	Q	M
Aluminum					
Antimony					
Arsenic					
Barium					
Beryllium					
Cadmium					
Calcium					
Chromium					
Cobalt					
Copper					
Iron					
Lead					
Magnesium					
Manganese					
Nickel					
Potassium					
Selenium					
Silver					
Sodium					
Thallium					
Vanadium					
Zinc					

9-IN METHOD DETECTION LIMITS (ANNUALLY)

			ntract:	
Lab Code:	Case No.:	NRAS No.:		SDG No.: _
Instrument Typ	pe:	Instrument ID:		Date:
Preparation Me	ethod:			
Concentration	Units (ug/L or mg	/kg):		
	F		1	
	Analyte	Wavelength /Mass	CRQL	MDL
		/ Mass		1
	Aluminum			
	Antimony Arsenic			
	Barium			
	Beryllium			
	Cadmium			
	Calcium			
	Chromium			
	Cobalt			
	Copper			
	Iron			
	Lead			
	Magnesium			
	Manganese			
	Mercury			
	Nickel			
	Potassium			
	Selenium			
	Silver			
	Sodium			
	Thallium			
	Vanadium			
	Zinc			
	Cyanide			
				_
			J	

10A-IN ICP-AES INTERELEMENT CORRECTION FACTORS (QUARTERLY)

Lab Name:					Contrac	t:	
Lab Code:		Case No	.:	NRAS N	o.:	SDG	No.:
ICP-AES Inst	rument	ID:		Date	:		
	1	I					
		Wave-		relement (Correctio	n Factors	for:
7~~	1,,,+0	length (nm)		Ca	Fo	Ma	
		(11111)	AI	Ca	ге	Mg	
Alum							
Anti							
Arse	nic						
Bari	um						
Bery	llium						
Cadm	ium						
Calc	ium						
Chro	nium						
Coba	lt						
Coppe	er						
Iron							
Lead							

Magnesium
Manganese
Nickel
Potassium
Selenium
Silver
Sodium
Thallium
Vanadium
Zinc

Comments:			

10B-IN ICP-AES INTERELEMENT CORRECTION FACTORS (QUARTERLY)

	'					t:	
Cod	e:	Case No.	•	NRAS N	o.:	SDG	No.:
AES	Instrument	TD:		Date	•		
1100	1110 et amerie				•		
				_			
		Wave-	Inter	relement (Correction	n Factors	for:
		length					
	Analyte	(nm)					
ſ	Aluminum						
	Antimony						
	Arsenic						
	Barium						
l	Beryllium						
	Cadmium	<u> </u>					
	Calcium						
	Chromium						
	Cobalt						
	Copper						
	Iron Lead						
ŀ	Magnesium	-					
ŀ	Manganese						
ŀ	Nickel						
ŀ	Potassium						
	Selenium						
	Silver						
ı	Sodium						
	Thallium						
	Vanadium						
	Zinc						

11-IN ICP-AES and ICP-MS LINEAR RANGES (QUARTERLY)

	Name:			Contract:	
b	Code:	Case No.:		NRAS No.:	SDG No.:
<u> </u>	Instrument I	D:		Date:	
			Integ. Time	Concentration	
		Analyte	(Sec.)	(ug/L)	M
		Aluminum			
		Antimony			
		Arsenic			
		Barium			
		Beryllium			
		Cadmium			
		Calcium			
		Chromium			
		Cobalt			
		Copper			
		Iron			
		Lead			
		Magnesium			
		Manganese			
		Nickel			
		Potassium			
		Selenium			
		Silver			
		Sodium			
		Thallium			
		Vanadium			
		Zinc			
		1		1	
		<u> </u>			

12-IN PREPARATION LOG

Lab Name:		Contract:	
Lab Code:	_ Case No.:	NRAS No.:	SDG No.:
Preparation Method			
EPA Sample No.	Preparation Date	Weight (gram)	Volume (mL)
		1	
	ĺ		

13-IN ANALYSIS RUN LOG

Lab Name:		Contract: _		
Lab Code:	Case No.:	NRAS No.:	SDG No.:	
Instrument I	D:	Analysis Meth	od:	
Start Date:		End Date:		

EPA														Aı	nal	yte	:S											
Sample No.	D/F	Time	A L	S B	A S	ВА	B E	C D	C A	C R	C O	C U	F E	P B	M G	M N	H G	N I	K	S E	A G	N A	T L	V	Z N	C N		
		ļ																										
	-																										-	\dashv
																											\dashv	\dashv
	<u> </u>	 																										=
	-				_											_												
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	1	1															I										ı	

14-IN ICP-MS Tune

%RSD
_

15-IN ICP-MS Internal Standards Relative Intensity Summary

Lab	Name:		Contract:	
Lab	Code:	Case No.:	NRAS No.:	SDG No.:
ICP-	MS Instrument]	ID:	Start Date:	End Date:

	_			Int	terna	l Standar	ds %1	RI For:						
EPA Sample No.	Time	Element	Q	Element	Q	Element	Q	Element	Q	Element	Q			

SAMPLE LOG-IN SHEET

Lab	Name		- 	100 110 51111			Page of							
Received By (Print Name) Log-in D														
Rec	Received By (Signature)													
Cas	e Number		Sample De	livery Grou	p No.		NRAS Number							
					Correspond	ing								
Rem	marks:		EPA Sample #	Aqueous Sample pH	Sample Tag #	Assigned Lab #	Remarks: Condition of Sample Shipment, etc.							
1.	Custody Seal(s)	Present/Absent* Intact/Broken												
2.	Custody Seal Nos	·												
3.	Traffic Reports/Chain of Custody Records or Packing Lists	Present/Absent*												
4.	Airbill	Airbill/Sticker Present/Absent*												
5.	Airbill No.													
6.	Sample Tags	Present/Absent*												
	Sample Tag Numbers	Listed/Not Listed on Traffic Report/Chain of Custody Record												
7.	Sample Condition	Intact/Broken*/ Leaking												
8.	Cooler Temperature Indicator Bottle	Present/Absent*												
9.	Cooler Temperature													
10.	Does information on Traffic Reports/Chain of Custody Records and sample tags agree?													
11. Date Received at Lab														
12.	Time Received													
	Sample I	ransfer												
Fra	ction	Fraction												
Are	ea #	Area #	<u> </u>											
Ву		Ву	1											
On		On]											
* (Contact SMO and at	tach record of resolution	lution											

FORM DC-1

Logbook No.

Reviewed By

Date

Logbook Page No.

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	FULL INORGANICS COMPLETE SD	G LIDE (C	JE / INVENTO	IXI OHEEL		
	LABORATORY NAME					
	CASE NO SDG NO					
	SDG NOs. TO FOLLOW					
	NRAS NO					
	CONTRACT NO.					
	SOW NO.					
	All documents delivered in the Complete SD where possible. (Reference - Exhibit B Se	G File mus	st be origi	nal docume	ents	
	ere peedaare, (Nererenee Zimaare 2 ee		NOs.	CHI	ECK	
1		FROM	<u>TO</u>	LAB	REGION	
1.	Cover Page					
2.	SDG Narrative					
3.	Sample Log-In Sheet (DC-1)					
4.	Inventory Sheet (DC-2))					
5.	Traffic Report/Chain of Custody Record(s)					
6.	<pre>Inorganic Analysis Data Sheet (Form I-IN)</pre>					
7.	<pre>Initial & Continuing Calibration Verification (Form IIA-IN)</pre>					
8.	CRQL Standard (Form IIB-IN)					
9.	Blanks (Form III-IN)					
10.	ICP-AES Interference Check Sample (Form IVA-IN)					
11.	<pre>ICP-MS Interference Check Sample (Form IVB-IN)</pre>					
12.	Matrix Spike Sample Recovery (Form VA-IN)					
13.	Post-Digestion Spike Sample Recovery (Form VB-IN)					
14.	Duplicates (Form VI-IN)					
15.	Laboratory Control Sample (Form VII-IN)					
16.	ICP-AES and ICP-MS Serial Dilutions (Form VIII-IN)					
17.	Method Detection Limits (Annually) (Form IX-IN)					
18.	ICP-AES Interelement Correction Factors (Quarterly) (Form XA-IN)					
19.	ICP-AES Interelement Correction Factors (Quarterly) (Form XB-IN)					

20. ICP-AES and ICP-MS Linear Ranges (Quarterly) (Form XI-IN)

21. Preparation Log (Form XII-IN)

			PAGE	PAGE NOs.		<u>CHECK</u>	
22.	Analysi	is Run Log (Form XIII-IN)	FROM	<u>TO</u>	LAB	REGION	
	1						
23.	ICP-MS	Tune (Form XIV-IN)					
	Intensi	Internal Standards Relative Lty Summary (Form XV-IN)					
		S Raw Data					
26.	GFAA Ra	aw Data (If Applicable)					
27.	ICP-MS	Raw Data					
28.	Mercury	7 Raw Data					
29.	Cyanide	e Raw Data					
30.	Prepara	ation Logs Raw Data					
31.	Percent	Solids Determination Log					
32.		Shipping/Receiving Documents rbill (No. of Shipments)					
	Sa	mple Tags					
	Sa	mple Log-In Sheet (Lab)					
33.		Shipping/Receiving Records					
	Te	lephone Logs					
34.		al Lab Sample Transfer Records & ng Sheets (describe or list)					
							
35.	Analysi	al Original Sample Prep & Ls Records (describe or list) ep Records					
		alysis Records					
		scription					
36.		Records (describe or list)					
		lephone Communications Log					
37.	Comment	::					
	pleted k P Lab)	ey:					
(СЦ		(Signature)	(Print Name	& Title)		(Date)	
	ited by: EPA)	-		<i>,</i>		· · · ·	
	•	(Signature)	(Print Name	& Title)		(Date)	

(Print Name & Title) FORM DC-2-2

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EXHIBIT C

INORGANIC TARGET ANALYTE LIST WITH CONTRACT REQUIRED QUANTITATION LIMITS

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EXHIBIT C - INORGANIC TARGET ANALYTE LIST WITH CONTRACT REQUIRED QUANTITATION LIMITS

Table of Contents

Section	<u>on</u>	Page
1.0	INORGANIC TARGET ANALYTE LIST AND CONTRACT REQUIRED	
	QUANTITATION LIMITS (CRQLs)	. 5

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1.0 INORGANIC TARGET ANALYTE LIST AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQLs)

Analyte	CAS Number	ICP-AES CRQL for Water ^{1,2,3,4} (μg/L)	ICP-AES CRQL for Soil ^{1,2,3,4,5} (mg/kg)	ICP-MS CRQL for Water ^{1,2,4} (μg/L)
7. 7	7400 00 5	200	2.0	
Aluminum	7429-90-5	200	20	
Antimony	7440-36-0	60	6	2
Arsenic	7440-38-2	10	1	1
Barium	7440-39-3	200	20	10
Beryllium	7440-41-7	5	0.5	1
Cadmium	7440-43-9	5	0.5	1
Calcium	7440-70-2	5000	500	
Chromium	7440-47-3	10	1	2
Cobalt	7440-48-4	50	5	1
Copper	7440-50-8	25	2.5	2
Iron	7439-89-6	100	10	
Lead	7439-92-1	10	1	1
Magnesium	7439-95-4	5000	500	
Manganese	7439-96-5	15	1.5	1
Mercury	7439-97-6	0.2	0.1	
Nickel	7440-02-0	40	4	1
Potassium	7440-09-7	5000	500	
Selenium	7782-49-2	35	3.5	5
Silver	7440-22-4	10	1	1
Sodium	7440-23-5	5000	500	
Thallium	7440-28-0	25	2.5	1
Vanadium	7440-62-2	50	5	1
Zinc	7440-66-6	60	6	2
Cyanide	57-12-5	10	2.5	

 $^{1}\mbox{The CRQLs}$ are the minimum levels of quantitation acceptable under the contract Statement of Work (SOW).

 $^2\mathrm{Subject}$ to the restrictions specified in Exhibit D, any analytical method specified in ILM05.3 Exhibit D may be utilized as long as the documented Method Detection Limits (MDLs) are less than one-half the CRQLs.

 $^3\mathrm{Mercury}$ is analyzed by cold vapor atomic absorption. Cyanide is analyzed by colorimetry/spectrophotometry.

 4 Changes to the Inorganic Target Analyte List (TAL) (e.g., adding an additional analyte) or CRQLs may be requested under the modified analysis clause in the contract.

 $^5 The$ CRQLs for soil are based on 100% solids and on the exact weights and volumes specified in Exhibit D. Samples with less than 100% solids may have CRQLs greater than those listed in the table above.